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A LABORATORY
OUTLINE OF
ELEMENTARY
CHEMISTRY

ALEXANDER SMITH

GENERAL DIRECTIONS.

Note Book. Enter the number and title of the exercise, and also the letter (*e.g.*, a).

Do not copy the printed directions. State in your own words the object of the experiment, what you did, what you observed, and your conclusions. Make a drawing of the apparatus.

Every interrogation point (?) calls for the entry of an observation, or an equation or other note. Answer also every direct question.

Numbers, such as [60], refer to sections in the Author's "Text-book of Elementary Chemistry," which must be read.

Quantities of materials, including solids, to be judged by the eye are stated in c.c. For quantities stated in grams, use the laboratory scales.

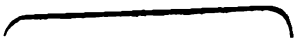
Do not take more than the amount specified. There are always good reasons for limiting the amount to that mentioned.

The stopcock is intended for regulating the size of the Bunsen flame. A small flame is usually sufficient, and a large one often breaks the vessel, or over-heats the material.

Solids, such as matches, filter-papers, and unconsumed chemicals, must be thrown into the waste jars, and not into the sink.

Noxious gases and vapors are indicated by the word [HOOD]. Perform such experiments in the hood or provide otherwise for good ventilation.

Always use clean vessels, otherwise misleading observations will be made and much time wasted.





**A LABORATORY OUTLINE
OF
ELEMENTARY CHEMISTRY**

A Companion Text to this Outline

**A TEXT-BOOK OF
ELEMENTARY CHEMISTRY**

BY
ALEXANDER SMITH

12 mo. 438 pages. With 6 plates
and 98 figures

\$1.25

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A Laboratory Outline OF Elementary Chemistry

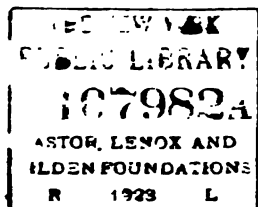
BY

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of Chemistry of Columbia University



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1918



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PREFACE.

In preparing this Outline, several things have been kept in view. For example: The selection, the order, and the form of statement have been arranged so as to harmonize with, and suitably to illustrate the subject matter of the Author's "Text-Book of Elementary Chemistry."

The apparatus has been made as simple and small in amount as possible. The list of chemicals has also been restricted to the least expensive materials and the smallest number of items. The indispensable experiments which are less simple have been left for the class-room demonstrations.

All the experiments, and the wording of the directions, have been tried under the Author's supervision with young pupils, and have been altered until found satisfactory.

The lengths of the laboratory periods assigned to chemistry are so different in different institutions, varying from 45 minutes to 90 minutes and even more, that exercises arranged for any one length of period would still be a misfit in the majority of cases. To make adjustment easy, the exercises have been subdivided, so that where the whole exercise would occupy more than 45 minutes, certain parts may be omitted or assigned to the next period.

It is not expected that the whole of the work outlined will be done by any one class. A choice is required, so that the interests of the instructor, the sex of the pupils, and other factors may receive consideration.

One of the chief errors into which many students fall is that of doing the experiments blindly and mechanically. In an effort to prevent this, the object of each exercise is stated

PREFACE

at the top. The attention of the class should always be directed to this by asking a few questions, to see whether it has been read and understood, before the work begins.

To assist the pupil in preparing for the class-room discussion, the teacher should add a number of additional questions in connection with each exercise.

Special attention has been given to exercises involving review. This usually takes the form of practice in identifying unknown substances, a form of reviewing which always strongly awakens the interest of the pupil. The exercises wholly devoted to this end are indicated in Appendix VII, but the same feature is included in many of the others.

The exercises dealing with the identification of unknown substances have been restricted in each case to a specific list, so as to limit the possibilities and to exclude complications which would discourage the pupils at this stage. The purpose is not to teach qualitative analysis, but solely to afford a review of methods of preparation, of important reactions and properties, and of methods of reasoning characteristic of chemistry. Even with the limitations adopted, these exercises contain all the material that there is time fully to utilize.

The Author is indebted to many teachers who have in one way or another assisted him in the preparation of this Outline. He wishes particularly to express his indebtedness to Mr. Oscar R. Flynn of the High School of Commerce, New York, to Dr. R. P. Calvert and Professor H. C. Sherman of Columbia University, to Miss Frances Church of the East High School, Des Moines, and to Mr. N. Henry Black of the Roxbury Latin School, Boston. He will be glad to be notified of any points which are found to present difficulties.

ALEXANDER SMITH.

COLUMBIA UNIVERSITY,
July, 1914.

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EXERCISE 1.

SUBSTANCES AND PROPERTIES.

Object: * *To observe and record the properties of some substances.*

Apparatus: Test-tubes. Rack. Evaporating dish. Wire gauze.

Materials: 2 pieces each of white cotton cloth,** white mixed goods,** and woolen yarn. Sodium hydroxide (sol., 8:100). Eosin or Acid Green (sol., 0.5%). Sulphuric acid (conc.).

a. Place 15 c.c. of sodium hydroxide solution in the graduated cylinder, add 40 c.c. of water, and mix. Divide the liquid equally between three test-tubes, immerse the pieces of cotton, wool, and mixed goods, one in each tube, and set the tubes in the rack. Heat the contents of the tubes one after another to the boiling point, holding each by means of a piece of paper folded so as to give four thicknesses (Fig. 1). Then keep one above the flame, at the boiling point, but not boiling, for two minutes, then the second, then the third, then the first again and so forth. When the material in one of them has all dissolved, examine all three. Record the results as follows:

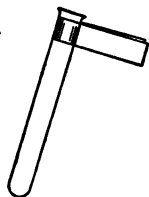


FIG. 1

DISSOLVED	IF CHANGED, How	APPEARANCE OF LIQUID
-----------	--------------------	-------------------------

Cotton:

Wool:

Mixed goods:

* Before every exercise, read this note on the object to be accomplished, and make sure that you understand it before proceeding.

** Before use, the cloth must be treated as in Ex. 73 a, to remove sizing.

b. Place the wire gauze on a ring on the ring stand (Fig. 2). Fill the evaporating dish three-fourths full of water, add two drops of concentrated sulphuric acid, place it on the gauze and heat to boiling. Add 10 c.c. of the solution of the dye provided. Immerse one sample each of the cotton, wool, and mixed goods, so that they are completely covered. Stir with the glass rod, and heat for 2 or 3 minutes. Pour the liquid into the sink and wash the materials thoroughly in clean water. Note the effect on each(!).



FIG. 2

c. If a microscope is available, place shreds of each sample, after drying, on a strip of glass and describe the appearance of the fibers as to structure(!) and color(!).

d. Record the specific properties of wool and of cotton, both those observed in these experiments and those already known to you: natural color(!), natural structure(!), solubility in water(!), solubility in sodium hydroxide solution(!), effect of dyes(!).

e. Smooth out the pieces of cloth and yarn on glass plates, or on bottles, to dry, and mount them in your notes.

f. Wash out the test-tubes and dish thoroughly and put them away. Throw scraps of cloth into the jars, and not into the sink. Cleaning up in this way follows every experiment hereafter.

EXERCISE 2.

CAPACITIES OF VESSELS.

Object: *To learn the capacities of several vessels by measuring the volumes of liquids they hold.*

Apparatus: Graduated cylinder. Test-tube. Evaporating dish.
Flask.

Materials: 2 small rubber bands.

a. Place water in the graduated cylinder up to the 5 c.c.

mark. The lowest point of the meniscus must be opposite the mark (Fig. 3). Pour this water into a test-tube and place a

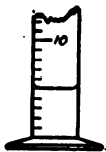


Fig. 3

narrow rubber band round the test-tube at the level of the lowest point of the meniscus. Measure and place in the test-tube a second 5 c.c. of water. Mark with a second rubber band the new position of the meniscus. Make in your notebook a full-size drawing of the test-tube, and show the positions of the bands. Observe carefully the proportions of the tube occupied by 5 c.c. and by 10 c.c. of water, so that when directed in future to use, say, "about 10 c.c." of a liquid or a solid you may be able to judge the amount by the eye.

b. Fill the graduated cylinder with water to the highest mark. Fill the evaporating dish with water from the cylinder, note the volume of water remaining, and subtract to learn the capacity of the dish. Record all the figures in your notes. Repeat with the flask.

c. What is the weight of 1 c.c. of water [p. 422]? What weights of water do the evaporating dish and the flask hold?

EXERCISE 3.

WEIGHING AND DENSITY.

Object: *To learn to weigh and to learn how to measure densities.*

Apparatus: Balance. Evaporating dish. Graduated cylinder. 20 c.c. pipette (one for every 10-20 pupils).

Materials: Roll sulphur. Sodium chloride (sat. sol.).

a. Weigh the evaporating dish (dry) on the balance to the nearest decigram (0.1 g.) and record the weight. In doing this, record first the weights missing from the box. Then record the weights on the pan of the balance, and note whether the numbers agree. Finally, re-count the weights to make

sure that no error has been made. In all weighings with the balance, count the weights three times, as just described.

Place in the dish about 10 g. of pieces of roll sulphur (no particles or dust of sulphur), weigh the whole once more and count the weights as directed above (record). The difference (?) is the exact weight of the sulphur actually taken.

Place in the graduated cylinder exactly 20 c.c. of water. Incline the cylinder and allow the sulphur gently to slide into water in the cylinder. Read the total volume (record). The increase (?) is the volume of the sulphur. Calculate from your data the weight of 1 c.c. of sulphur (?). This is the density of the sulphur.

b. Record the specific physical properties of sulphur which you have observed, namely: color (?), crystalline or not (?), brittle or malleable (?), density (?), soluble in water or not (?).

c. Wipe out the evaporating dish, and use the weight already recorded. Fill a 20 c.c. pipette to the mark with a saturated solution of common salt, allow this solution to run gently into the dish, and weigh again (record). The difference (?) is the weight of the brine. Calculate the density of the brine (?).

EXERCISE 4.

CHANGES IN METALS HEATED IN AIR.

Object: *To observe changes in properties when a new substance is formed by the action of oxygen from the air.*

Apparatus: Balance. Porcelain crucible. Pipe-stem triangle. Meter stick (one for class). Piece of iron wire, No. 26.

Materials: Copper wire (No. 30). Tin foil.

a. Take about 5 meters of copper wire (no. 30), wind it round a pencil, and place the coil in the porcelain crucible. Weigh the whole on the balance, counting the weights as in 3 a (record). Place the crucible (uncovered) in the pipe-stem

triangle(Fig. 4) and heat, at first gently and then with the full Bunsen flame, for fifteen minutes. Now lower the flame to permit the crucible to cool gradually, and finally remove it. Weigh the crucible when cold(record). To what is the difference in weight due?

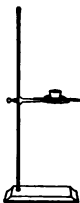


FIG. 4

Examine the coil(?), and bend the remains of the wire(?). Name the product, and make a condensed statement [18] of the change. Record the specific physical properties of the copper and of the product, namely: color(?), appearance(metallic or otherwise?), pliability or brittleness(?) of each.

b. Fold up about 10 cm. square of tin foil and place it in the porcelain crucible. Weigh the whole on the balance(record) and proceed as in a par. 1. Stir the tin occasionally with a short piece of iron wire, but be careful to keep all the material in the crucible. Describe what you observe(?).

Name the product(?) and make a condensed statement [18] of the change. Record the specific physical properties of the tin and of the product, namely, color(?), appearance(?), pliability(?) of each, and melting-point of tin [637] (?).

EXERCISE 5.

MIXTURES AND COMPOUNDS.*

Object: *To learn how, by observing properties, to distinguish between mixtures and compounds.*

Apparatus: Magnet. Test-tubes. Funnel. Evaporating dish. Mortar. Trip scales. Bunsen.

Materials: Iron powder (by alcohol). Carbon disulphide [CAUTION: Extinguish Bunsen flame while using this]. Filter-paper. Hydrochloric acid(dil.). Sulphur.

a. **The Properties of Iron.** Place about 0.5 c.c. of iron

* If too long for the period, this exercise may be divided between two periods.

powder on a piece of paper. Examine it(?). Draw one pole of a magnet across the lower surface of the paper, noting the behavior of the iron(?).

Transfer half the iron to a dry test-tube, add 2 c.c. of carbon disulphide [CARE! Inflammable! Do not handle near a flame], and shake. Fold a filter-paper once, and then again at right angles to the straight edge. Open the paper as a cone, with a triple layer of paper on one side and a single layer on the other, and place it in a glass funnel (Fig. 5). Place the funnel in one ring of the iron stand, place the evaporating dish below the end of



FIG. 5

the stem, and pour the contents of the test-tube on to the paper. When the liquid has run through, open up the paper, so that the remaining liquid may evaporate. Place the dish in a draft, away from all flames, where its contents may evaporate also. While evaporation is going on, proceed with the operations described in the following paragraphs. When the paper and dish are dry, examine the residue on the paper(?) and draw the pole of the magnet across the lower surface of the paper(?). Examine the dish(?). Is iron soluble in carbon disulphide(?).

Place the other half of the iron in a test-tube, add a few drops of hydrochloric acid and note the odor(?) or absence of odor.

Record the properties of iron, namely: color(?), effect of magnet(?), solubility in carbon disulphide(?), effect of hydrochloric acid(?) and odor or not(?).

b. The Properties of Sulphur. Pulverize some sulphur in the mortar, and repeat(a) using about 1 c.c. of sulphur instead of the iron. Record the properties of sulphur, not already studied in Exercise 3, namely: soluble in carbon disul-

phide or not(?), shape of crystals(sketch?), effect of hydrochloric acid(?) and odor or not(?).

c. **A Mixture.** On a piece of paper weigh out 3 g. of pulverized sulphur and 5.5 g. of iron powder. Mix the powders as well as possible. Can the particles of the substances still be recognized or not? Pass the magnet underneath the paper(?). Place half the mixture in a test-tube, add 3-4 c.c. of carbon disulphide, shake, and filter into the evaporating dish. When the liquid has evaporated, examine the residues on the paper and in the dish(?). Why does the result show that the material was a mixture, and not a chemical compound [16]?

d. Place the remainder of the mixture from c in a dry test-tube, hold the test-tube by means of a piece of folded paper(Fig. 1, Ex. 1), and heat it gently in the Bunsen flame(?). When the test-tube is cold, place the lower end in the mortar, cover with a towel, and strike through the towel so as to break it. Use the pestle in this way until the material, and the fragments of glass to which the material is attached, can be placed in another test-tube. Add 2 c.c. of carbon disulphide, filter, and allow the filtrate to evaporate as before. Examine the material on the filter to ascertain whether it is iron(?). If it is in the least affected by the magnet; too little sulphur was used. Does the filtrate yield all the sulphur used(?), or any sulphur? Place the material on the filter in a test-tube, add a few drops of hydrochloric acid (odor ?). Did either iron or sulphur give this odor?

Record the properties of ferrous sulphide, namely: color(?), effect of hydrochloric acid(?) and odor or not(?). What was the effect of heating the mixture? Is the product an element, or a compound, or a mixture?

EXERCISE 6.

OXYGEN—PRELIMINARY.

Object: *To learn which substances yield oxygen, and how to recognize it.*

Apparatus: Test-tube (hard glass). Clamp. Porcelain crucible. Dropper.

Materials: Lead dioxide. Wooden splints. Sand. Potassium chlorate. Manganese dioxide (pulv.). Sodium peroxide. Litmus papers.

a. Place about 1 c.c. of lead dioxide (properties?) in a dry hard glass test-tube, and fasten the tube in a vertical position in the clamp on the iron stand. Heat at first gently and then strongly with the Bunsen flame. Introduce a wooden splint, glowing at one end, into the tube until the spark almost reaches the oxide(?). What gas is liberated? What physical and chemical properties do you observe the gas to have? After removing from the flame, examine the residue in the tube(?). The new substance is litharge. Name the variety of chemical change illustrated [33].

b. Repeat a, using 1 c.c. of silicon dioxide (sand), and answer the same questions.

c. Repeat a, using 1 c.c. of potassium chlorate (properties?), and answer the same questions.

d. Repeat a, using 1 c.c. of manganese dioxide (properties?), having previously dried this by warming it in the porcelain crucible. Answer the same questions.

e. Place in a dry test-tube about 0.5 c.c. of sodium peroxide. Using a dropper, add one or two drops of water to the peroxide(?). Test this gas with a glowing splinter of wood(?). Afterwards, dip a piece of red litmus paper in the residual liquid(?). A blue color indicates the presence of an alkali [191] (sodium hydroxide).

EXERCISE 7.

GLASS-WORKING.

Object: To learn how to make simple apparatus.

Apparatus: Triangular file. Rack. Wing-top. Test-tube (hard glass) and stopper (1-hole).

Materials: Glass rod. Glass tubing.

a. Glass rod. Make a transverse notch about 15 cm. (6") from the end of a glass rod. To do this, hold the rod on the table and draw the edge of the triangular file once firmly and smoothly across it. Now, break the rod at this point by holding it so that the points of the thumbs are together on the side opposite to the notch, and pressing forward with the thumbs so as to bend the rod away from the mark. Cut two other pieces of the same length.

To remove the sharp, often jagged edges, hold the ends of the rods in the Bunsen flame, turning them slowly until the edges are rounded (fire-polished). Observe the color of the flame (?). To what is this color due [457]? Do not lay the hot rods on the table, but balance them across the test-tube rack or on an iron ring until cold. Why does heating the rods remove the sharp edges?

b. Glass tubing, drawing out. Cut, exactly as in **a**, from a piece of glass tubing a portion 15 cm. long. Hold the center of this piece of tubing in the Bunsen flame, turning it slowly, until it becomes soft (Fig. 6). Hold and turn it, as a whole, carefully, so as not to bend or

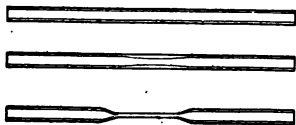
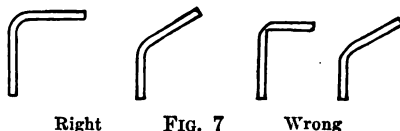


FIG. 6

twist it. When it is quite soft, remove it from the flame, and draw the two ends slowly apart.

c. Glass tubing, bending. Fit a wing-top on the Bunsen burner, and use the luminous flame. Hold a piece of tubing

15 cm. long in the flame so that as long a portion is heated as possible, and rotate it steadily so as to heat all sides alike. Do not allow the tube to bend or twist. When the glass is quite soft, withdraw the tube and bend it to form a right angle, giving an L-tube (Fig. 7).



The bend must be gradual and smooth, not sharp or crumpled. Bend another similar piece to form an obtuse angle. Fire-polish the edges (for this, use the non-luminous flame), but do not heat too long, otherwise the bore will be diminished.

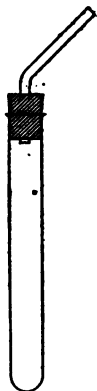


FIG. 8

d. Fit a stopper into the hard glass test-tube. Remove the stopper, and push the shorter end of the obtuse-angled tube through the hole (Fig. 8). Do not use force—moisten the tube and the hole and rotate the tube while pushing it gently through (don't hurry). Allow the tube to project not over 1 mm. on the inside. Replace the stopper in the test-tube and test for air-tightness. To do this, place the end of the tube in the mouth, suck out some of the air, and note whether the tip of the tongue seems to adhere to the tube. Remove the tube and stopper, and repeat(?). What caused the tongue to adhere in the first instance?

EXERCISE 8.

OXYGEN—PREPARATION AND PROPERTIES.

Object: *To make a quantity of oxygen and to ascertain its physical and chemical properties.*

Apparatus: Mortar. Trip scales. Test-tube. Test-tube (hard glass) with stopper and bent tube (Ex. 7 d). Narrow rubber tube 12'

long. Clamp. Pneumatic trough. 3 w.-m. bottles. 3 glass plates. Deflagrating spoon. Iron wire.

Materials. Potassium chlorate. Manganese dioxide(pulv.). Splints. Asbestos paper. Sulphur. Red phosphorus. Charcoal(splinters).

a. Pulverize 4 g. of potassium chlorate. Mix it on paper with 2 g. of manganese dioxide. Place about one-tenth of the mixture in a dry test-tube, heat it, and test the gas with a glowing splinter of wood. If flashes of light are seen during the heating, call the instructor's attention to them at once, and do not use the same mixture in b. Small sparks, due to dust, may be ignored.

b. Place the remainder of the mixture in the hard glass test-tube provided with a stopper and glass tube(Ex. 7 d), and test for air-tightness. Moisten the end of a piece of narrow

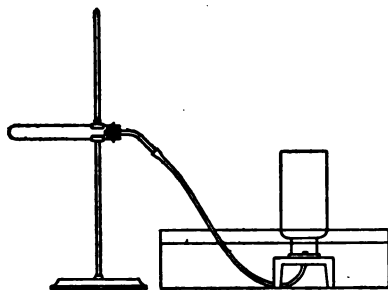


FIG. 9

rubber tubing(12" long) and slip it over the end of the tube. Clamp the tube in a horizontal position (Fig. 9). Fill the pneumatic trough with water so as to cover the shelf or other support. Fill 3 wide-mouth bottles with water, cover them with glass plates, and invert them,

one on the shelf, and the other two in the trough. Use a small Bunsen flame to heat the tube and collect the first bubbles of gas in a test-tube filled with water and inverted over the exit of the rubber tube. Test this gas with a glowing splinter(?). What is this gas, and where did it come from? Now fill the bottles one by one, covering each when full with a glass plate, and setting it mouth upwards on the table. During the operation, regulate the flame with great care so that the gas comes off in a steady, but not too rapid

stream of bubbles. If the gas begins to come too fast, move the flame promptly to another part of the material and lower it slightly. The glass must not become hot enough to tinge the Bunsen flame yellow. Do not remove the flame, however, at any time, without first taking the rubber tube out of the water (Why?). After the third bottle is filled, remove the rubber tube from the water, let the test-tube cool and then fill it with water and set it aside.

c. Line the deflagrating spoon with asbestos paper and place on the asbestos a little sulphur. Heat the sulphur until it catches fire. Observe the flame for a moment (?). Then lower the spoon into one bottle of oxygen (?), keeping the mouth as far as possible covered with the glass plate. How does the flame differ from that in air, and why? Is it finally extinguished? If so, why? Cautiously note the odor (?). Name the product (?) and state its physical properties (?).

d. Reline the spoon with asbestos and place on it a little red phosphorus. Set fire to the latter, observe the flame (?) and lower the spoon into the second bottle as before (?). Name the product (?) and state its physical properties (?).

Before putting the spoon away, heat both it and the asbestos strongly in the Bunsen flame to burn up all the phosphorus.

e. Wrap the end of a piece of wire round a splinter of charcoal, set fire to the latter, and lower it into the third bottle as before (?). How does its combustion compare with that in air? Name the product (?) and state its physical properties (?).

f. Record the physical properties of oxygen: color (?), odor (?), solubility in water (?). Record the chemical properties: glowing splint (?), sulphur (?), phosphorus (?), charcoal (?). What is the difference between a chemical and a physical property? *

* Exercise 14 (except f) may be introduced here, if desired.

EXERCISE 9.

HYDROGEN—PRELIMINARY.

(Interaction of metals with acids and water.)

Object: To learn various ways of obtaining hydrogen and to compare them.

Apparatus: 12 test-tubes. Graduated cylinder. Evaporating dish. Large beaker.

Materials: Iron (nails). Zinc (gran.). Tin (gran.). Copper (shavings). Aluminium (wire). Magnesium (ribbon). Hydrochloric acid (conc.). Zinc (dust). Sulphuric acid (dil.). Acetic acid. Calcium.*

a. Place in separate test-tubes a few small pieces of each of the metals, iron (nails), zinc (granulated), tin (granulated), copper (shavings), aluminium (wire), magnesium (ribbon). Pour into the graduated cylinder 20 c.c. of pure concentrated hydrochloric acid, add an equal volume of water, and mix. Add 5 c.c. of this diluted acid to the contents of each tube.

Observe each case critically, and tabulate the results. Is there bubbling? If not, then warm gently(?). If the eye detects the formation of a gas or vapor, smell the contents of the tube(?). For comparison, take 5 c.c. of the acid by itself in a test-tube, smell it(?), warm and smell again(?). If heating seems to produce a gas, remember that it may be the acid, or steam and not hydrogen. The formation of hydrogen may be inferred from continued bubbling when heat is not being furnished, and may be proved by the slight explosion which follows when a light is brought to the mouth of the tube. If the gas is coming slowly, hinder its escape into the air by partially closing the mouth of the tube and let it accumulate before applying the light.

Number the metals in your notes in the order of their ap-

* Calcium (metal) is most easily broken into small pieces by fixing it in a vise and using a chisel and hammer.

parent activity (most active = No. 1). Do the bubbles appear on the side of the glass tube or on the metal? Why do they appear on one and not on the other? Record the properties of hydrogen you have observed: Gas, liquid or solid(?), soluble in water or not(?), colored(?), odor or not(?), unites with oxygen from the air or not(?) and if so when cold or hot(?).

b. After the action has ceased, filter any one of the solutions and evaporate (Ex. 10 f) 1 c.c. of it to dryness [HOOD] (?). What is the product? Record the action in each case in the form of a condensed statement [18].

c. Add some zinc dust to the remaining 5 c.c. of the acid(?). To what is the difference, if any, between the apparent activity of zinc dust and granulated zinc due?

d. The same metals displace hydrogen from all acids. To illustrate, place a few pieces of zinc (gran.) in each of two test-tubes and add to one 5 c.c. of dilute sulphuric acid(?) and to the other 5 c.c. of acetic acid(?).

e. Fill a test-tube with water, and invert it in the large beaker half filled with water. Place a small piece of calcium in the water and hold the test-tube over it(?). After the action has ceased, apply a light to the gas in the tube(?). Examine the water in the beaker(?) and test it with red litmus paper(?).

EXERCISE 10.

HYDROGEN—PREPARATION AND PROPERTIES.

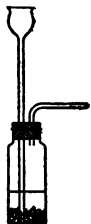
Object: To prepare a quantity of hydrogen and observe its physical and chemical properties.

Apparatus: 90 c.c. gas-generating bottle with 2-hole stopper and thistle-tube. Glass tubing. 12" narrow rubber tube. Trough. Trip scales. Test-tubes. 2 w.-m. bottles and 2 glass plates. Watch glass. Beaker. Wire gauze. Taper.

Materials: Zinc (gran.). Sulphuric acid (conc.). Cupric oxide.

a. Fit the 90 c.c. gas-generating bottle with a 2-hole stop-

per, a thistle-tube, and an L-tube (Fig. 10). Slip the narrow rubber tube over the end of the last. Place in the bottle about 20 g. of zinc (gran.). Test the apparatus to see whether it is air-tight. To do this, pour into the bottle through the thistle-tube enough water to cover the zinc. The lower end of the thistle-tube must also be under the water. Now blow through the rubber-tube a little air, so as to force the water up the stem of the thistle-tube, and immediately compress the rubber-tube with the thumb and fore-finger. If the water does not remain stationary, but



gradually falls, there is a leak which must be remedied [Instructor].

Add concentrated sulphuric acid slowly through the thistle-tube until brisk bubbling sets in. Do not add more than one-fourth of the volume of the water already in the bottle. Place the end of the rubber-tube in the trough. Collect a test-tube full of the gas. Close the tube with the thumb and carry it mouth downwards to a distant flame. Remove the thumb and, still holding the tube mouth downwards, set fire to the gas. Repeat this test until a sample is obtained which burns quietly. Why does it not do so at first? Do not at any time attempt to light the gas at the exit tube.

b. Collect over water two bottles of the gas. Cover the first with a glass plate and set it mouth downwards on the table. Light a taper, raise this bottle (still mouth downwards), insert the burning taper (?) almost to the bottom of the bottle (?) and then withdraw it slowly (?). What happens to the gas? What happens to the taper on insertion and on withdrawal? Explain.

c. Set the other bottle mouth upwards on the table and leave it open for one minute. Then bring a lighted taper to the mouth (?). Explain.

d. Fill a test-tube over water with the gas. Bring this

tube, mouth downwards, mouth to mouth with a test-tube filled with air, and keep them in this position for 3 minutes. Then apply a light first to the lower(?) and then to the upper tube(?). What fact about the molecules of gases is shown by this experiment?

e. Place 0.5 c.c. of cupric oxide in the bottom of a dry test-tube and fasten the latter in a horizontal position in the clamp on the iron stand. Insert a straight piece of glass tubing (ends fire-polished) into the rubber delivery tube and introduce this tube into the test-tube until the end touches the cupric oxide. Wait 3 minutes, to permit the air to be replaced by hydrogen. If the hydrogen is being given off too slowly, pour a little more sulphuric acid into the thistle-tube. Now heat the cupric oxide with a Bunsen flame. Note any change in the cupric oxide(?) and anything appearing in or on the cool part of the tube(?).

Make a condensed statement of this action. What kind of chemical action is this [67]?

f. Place 3 drops of the liquid from the gas-generating bottle on a watch glass. Place the watch glass on a beaker half filled with water, set the beaker on the wire gauze and boil the water. If the water in the beaker runs too low, because of evaporation, the vessel may crack. Add more hot water, if necessary. What is the solid left on the watch-glass by evaporation?

g. Record the observed physical properties of hydrogen, namely: color(?), odor(?), density compared with air(?); also two chemical properties observed in b(?) and one in e(?).

EXERCISE 11.

WATER—PHYSICAL PROPERTIES.

Object: *To learn how to purify water, and how to find out whether it contains non-volatile impurities*

Apparatus: Flask. Glass tubing. Test-tubes. Beakers. Clamp. Wire gauze. Watch glass.

Materials: Marble(chips). Potassium permanganate(sol.). Phenolphthalein(sol. in alcohol, 1:500). Ammonium hydroxide(sol.).

a. (Two pupils working together.) Put on a clean watch-glass 3-4 drops of distilled water, place the watch-glass on a 100 c.c. beaker half filled with water, and set the beaker on the wire gauze and boil(see directions in Ex. 10 f). The second pupil uses 3-4 drops of tap-water in the same way. Compare the two watch glasses when dry(?). Do the specimens of water contain any non-volatile impurities?

b.* **Distillation.** Bend a piece of glass tubing twice at right angles and fire-polish the ends. Connect with it, by means of a short piece of rubber tubing, a straight tube about 30 cm. long. Set up a flask and a test-tube immersed in cold water in the 300 c.c. beaker as in Fig. 11. Place in the flask about 100 c.c. of water, some marble chips(to prevent "bumping"), and a few drops of potassium permanganate solution. Boil the water and examine the distillate(in the test-tube).

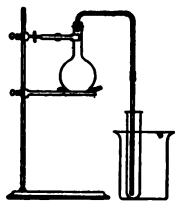


FIG. 11

In what way has the water been purified by the distillation? Is potassium permanganate volatile under these conditions? If it had been, would distillation have purified the water?

c. Add one drop of phenolphthalein solution to some tap-water(?). Add one drop of ammonium hydroxide solution to some tap-water, and then add one drop of phenolphthalein(?).

d. Using a glass rod, add a single drop of ammonium hydroxide solution to 100 c.c. of tap-water. Clean and use the same apparatus as in b, place this water in the flask, and distil as before. In each of six clean test-tubes place, from

*To save time, one pupil of a pair may do b while the other does c and d.

a glass rod, one drop of phenolphthalein solution, and use these test-tubes one after another to catch the distillate. Change the test-tube when 10 c.c. of liquid has come over until the six have been used. While changing, remove the flame and uncork the flask each time. If the water in the beaker becomes warm, change it for cold water.

What evidence is there that ammonia passes over with the steam? Which portion of the distillate contained the most ammonia and which the least? Cool the residue in the flask in running water, pour 10 c.c. of it into a clean test-tube and add one drop of phenolphthalein(?). Can water be purified from a volatile impurity?

e. Name the common states in which water exists(?). How many states of water are known altogether [75]?

EXERCISE 12.

HYDRATES.

Object: *To learn how to find out whether a substance is a hydrate or not.*

Apparatus: Watch glass. Test-tubes. Clamp. Mortar.

Materials: Washing soda. Potassium chlorate. Aluminium sulphate. Potassium sulphate. Barium chloride. Gypsum (fragments). Blue-stone.

a. Place a clear crystal of washing soda (sodium carbonate) on a watch glass and set it aside. At the end of the period, examine it and record any changes you observe(?).

b. Place a clear crystal of washing soda in a test-tube and heat the crystal gently(?). What condenses on the walls of the tube? Explain the result of a [81].

c. Place in as many dry test-tubes about 1 c.c. each of potassium chlorate, aluminium sulphate, potassium sulphate, barium chloride, and gypsum (calcium sulphate). Clamp one tube in a horizontal position, to prevent any condensed water

from running back and causing the tube to crack, and heat the substance with the tip of a small flame until no further change occurs(?). Repeat with each substance. Record the results in tabular form as follows:

SUBSTANCE HEATED	WHAT CHANGE	AMOUNT OF CONDENSED WATER	APPEARANCE OF FINAL RESIDUE	SUBSTANCE WAS A HYDRATE OR NOT
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The amount of the water condensed may be "great," "small," or "a trace." Remember that even an anhydrous substance may contain a little water, as an impurity. Hence, a substance is not to be classed as a hydrate(last column) unless the liberation of water is distinctly evident.

Are all crystalline substances hydrates? Illustrate. Make a condensed statement [18] of the change in each case in which water was given off.

d. Take about 1 c.c. of blue-stone(cupric sulphate). Note its color(?). Pulverize it finely in the mortar and note the color again(?). Explain.

Place the powder in a dry test-tube, clamp the latter in a horizontal position, heat the substance as in c and tabulate the results as before.

Leave the tube in the clamp until it is cold, and then set it upright(?). If there is not enough water condensed to permit some to run down to the solid residue, add two drops (?). Note the color. What substance is formed?

Make a condensed statement, showing the action to be reversible [83]. Record its color under the name of each substance.

EXERCISE 13.

MEASUREMENT OF WATER OF HYDRATION.

Object: To find the per cent of the elements of water in gypsum(hydrated calcium sulphate) or in barium chloride(quantitative).

Apparatus: Porcelain crucible. Balance. Pipe-stem triangle.

Materials: Gypsum (crystal, pulv.). Barium chloride.

a. Clean and dry the porcelain crucible and weigh it to the nearest centigram (0.01 g.), counting the weights as in 3 a. Then place in it about 2 g. of pulverized gypsum (do not try to take this exact amount) and weigh again as before. Record the weights in tabular form as shown below.

Support the crucible (open) on the pipe-stem triangle, placed on the ring of the iron stand, so that the bottom of the crucible is a short distance above the inner cone of the Bunsen flame. Lower the flame and heat at first very gently to avoid loss of any particles by sudden splitting of the crystals. Later use the full flame.

After 15 minutes' heating, allow the crucible to cool and weigh to the nearest centigram. Replace the crucible on the triangle, heat again for 5 minutes, allow to cool and weigh again. If the weight is less than before, heat once more, and repeat until two successive weighings are identical, *i.e.* "heat to constant weight." What does this constant weight show about the water of hydration?

Wt. of crucible + gypsum	g.
Wt. of crucible empty	g.
Wt. of gypsum taken	<hr/> g.
Wt. after first heating	g.
Wt. of crucible + gypsum	g.
Wt. after last heating	g.
Wt. of water of hydration	<hr/> g.

$$\text{Per cent of water} = \frac{\text{wt. of water} \times 100}{\text{wt. of gypsum}} = ?$$

When the percentages obtained by all who have done the experiment are compared, what law of chemistry is found

to be illustrated? Record the average of the values found by all for the percentage.

b. Some members of the class may be directed to use barium chloride, following otherwise all the directions given for gypsum.

EXERCISE 14.

WEIGHT OF 22.4 LITERS OF OXYGEN.

Object: *To learn the weight of a measurable volume of oxygen. Also to make the necessary corrections and calculate the density and molecular weight of this, or of any other gas (quantitative).*

Apparatus: Trip scales. Porcelain crucible. Iron wire. Hard glass test-tube with one hole stopper, bent tube and narrow rubber tubing. 2-liter bottle. Trough. Thermometer (one for room). Barometer. Glass plate or cork. Balance. 500 c.c. graduated cylinder (1:10 pupils).

Materials: Manganese dioxide (pulv., dried). Potassium chlorate (C.P. pulv., dry).

a. Place 6 g. of pulverized manganese dioxide in the porcelain crucible (uncovered) and heat it with the full Bunsen flame for 5-6 minutes. During this time, stir it occasionally with an iron wire.

While this is going on, insert an obtuse-angled tube in a one-hole rubber stopper which fits the hard glass test-tube (as in Fig. 8, Ex. 7 d). Fill a 2-liter bottle with water, invert it in the pneumatic trough, and arrange the apparatus as in Fig. 9 (Ex. 8).

Take 7 g. of dry, pulverized potassium chlorate and, when the manganese dioxide has cooled, mix the two substances thoroughly on a sheet of paper. Place the mixture in the hard glass test-tube. Weigh on the balance the test-tube and contents to the nearest centigram and record the result according to the table below (?). Replace the stopper in the test-tube, and test for airtightness (See Ex. 7 d).

b. Heat the contents of the test-tube, beginning at the end next to the stopper, and collect all the gas in the bottle. Regulate the heating so that you can at all times count the bubbles. Continue heating until gas ceases to come off, or until there remains only time enough to make one weighing, and then remove the rubber tube from the water and allow the test-tube to cool.

Lower the bottle of gas in the pan until, when the eye is on a level with the water, the latter is seen to be at the same height inside and outside. To accomplish this and the next operation, it may be necessary to incline the bottle. While the bottle is in this position, close it with a cork or glass plate and set it mouth upwards upon the table. Read and record the temperature of the water (?) and also the barometric pressure(?). If, at this point, there is not time to complete the experiment, the test-tube may be corked tightly and put away (upright) along with the 2-liter bottle and its contents.

c. Weigh on the balance the test-tube and contents, and record the weight(?). To measure the volume of gas collected in the bottle, fill the 500 c.c. graduated cylinder to the top of the graduation, note down the volume of water, and pour the latter into the bottle. Repeat until the bottle is filled level with the mouth, and record in the table the total volume of water used.

Wt. of test-tube and contents before heating	g.
--	----

Wt. of test-tube and contents after heating	g.
---	----

Wt. of Oxygen	g.
---------------	----

Volume of oxygen	c.c.
------------------	------

Temperature	C
-------------	---

Barometric Pressure	mm.
---------------------	-----

Tension of aqueous vapor [Appendix I or p. 423]	mm.
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Barometric pressure, corrected	mm.
--------------------------------	-----

d. Reduce the observed volume of oxygen from the observed temperature and pressure to 0° and 760 mm.

e. From this reduced volume, and the weight, calculate the weight of 1 c.c. (the density) of oxygen [102]:

$$\frac{\text{Wt. of ox.}}{\text{vol. at 0° and 760 mm.}} = x = \text{wt. of 1 c.c.} = \text{density.}$$

f. Calculate also the weight of 22.4 liters (the molecular weight) of oxygen [101]:

$$\text{wt. of 1 c.c.} \times 22400 = x = \text{wt. of 22.4 l.} = \text{mol. wt.}$$

Compare the molecular weight found with the atomic weight of oxygen (?). How many atoms of oxygen are there in one molecule of the element?

EXERCISE 15.

ATOMIC AND MOLECULAR WEIGHTS.

Object: To find atomic weights from data given. Also to become familiar with the relations between density and molecular weight.

a. The weights of one G.M.V. (22.4 liters at 0° and 760 mm., 87-88) of several compounds of carbon are as follows:

	Wt. 22.4 l.	Wt. carb.	Wt. hyd.	Wt. ox.	Formula
Carbon monoxide	28.00	12		16	
Carbon dioxide	44.00	12		32	
Methane	16.032	12	4.032		
Ethylene	28.032	24	4.032		
Glycerine (vapor)	92.064	36	8.064	48	

What value should you select for the atomic weight of carbon [89]? Assuming the formulæ and atomic weights to be, for hydrogen H = 1.008, for oxygen O = 16, and for carbon C = ?, write the formulæ for each of the five compounds.

b. The density (wt. of 1 c.c. at 0° and 760 mm.) of ethylene chloride is 0.00433 g., what is the molecular weight [102]?

24 COMPOSITION OF NICKEL SULPHIDE [Ex. 16]

c. Using the weights in 22.4 l. given above, find the weight of 1 c.c.(the densities) of methane and carbon dioxide.

d. How do the densities of the five substances mentioned in a compare with that of air [102] ?

e. The weight of zinc combining with 35.46 g.(one atomic weight) of chlorine is 32.68 g. The specific heat of zinc (metal) is 0.0936. What is the atomic weight of zinc [105] ?

EXERCISE 16.

COMPOSITION AND FORMULA OF NICKEL SULPHIDE.

Object: To find the composition and formula of nickel sulphide (quantitative).

Apparatus: Porcelain crucible and cover. Balance. Pipe-stem triangle.

Materials: Nickel, powder ("reduced"). Flowers of sulphur.

a. Weigh the crucible(without cover) to the nearest centigram, counting the weights as in 3 a. Place about 2 g. of reduced nickel(do not attempt to take this amount, exactly) in the crucible and weigh again. Put about 2 g. of sulphur in the crucible, stir the contents, and place the crucible on the pipe-stem triangle. Set the cover on the crucible, and heat gently so long as sulphur vapor burns [HOOD] at the chink between the crucible and cover. Heat strongly for 1-2 minutes. Then hold the flame against the upper portion of the crucible, so that every part receives a thorough heating. Allow the crucible to cool. When it is cold(not before) remove the cover and weigh. Tabulate the results as follows:

Wt. of crucible + nickel	g.
Wt. of crucible empty	g.
Wt. of nickel taken	g.
Wt. of crucible + nickel sulphide	g.
Wt. of crucible + nickel	g.
Wt. of sulphur combined	g.

Wt. of crucible + nickel sulphide	g.
Wt. of crucible empty	g.
Wt. of nickel sulphide	g.

- b. Calculate the percentage composition:

$$\text{Per cent sulphur} = \frac{\text{wt. of sulphur} \times 100}{\text{wt. of nickel sulphide}} = ?$$

$$\text{Per cent nickel} = \frac{\text{wt. of nickel} \times 100}{\text{wt. of nickel sulphide}} = ?$$

- c. Find the formula of nickel sulphide [109]. To do this, first obtain the atomic weights of nickel and sulphur from the table.

$$\text{Wt. of sulphur} \div \text{at. wt. of sulphur} = ? \text{ (factor 1)}$$

$$\text{Wt. of nickel} \div \text{at. wt. of nickel} = ? \text{ (factor 2)}$$

The combining proportion of sulphur to nickel found in the experiment is therefore:

$$\frac{\text{At. wt. sulphur} \times \text{factor 1}}{\text{At. wt. nickel} \times \text{factor 2}} = \frac{\text{S} \times \text{factor 1}}{\text{Ni} \times \text{factor 2}}$$

Divide above and below by factor 1. What is the formula?

- d. Make the equation for the union of sulphur and nickel to form nickel sulphide.

EXERCISE 17.

COMPOSITION AND FORMULA OF OXIDE OF TIN.

Object: To find the proportions of tin and oxygen in oxide of tin (stannic oxide) and the formula of the latter (quantitative).

Apparatus: Porcelain crucible. Balance. Pipe-stem triangle.

Materials: Tin foil (free from lead). Nitric acid (conc.).

- a. Weigh the crucible (without cover) (?). Place in it about 1 g. of tin foil (do not try to take exactly 1 g.) and weigh again (?). Record the weights in tabular form (see be-

low) Place the crucible on the pipe-stem triangle and add 5 c.c. of concentrated nitric acid. In doing this, pour the acid over every part of the metal. Hold the burner in the hand and warm the crucible with a very small flame, stopping for a moment if there is a tendency for any particles to be spattered out of the vessel. When the contents are dry, use a larger flame and heat for ten minutes. Weigh when cold (?)

The nitric acid oxidizes the tin. A brown gas, nitrogen tetroxide, and water are given off during the process.

Wt. of crucible + tin	g.
Weight of crucible, empty	g.
Wt. of tin taken	<hr/> g.
Wt. of crucible + oxide of tin	g.
Wt. of crucible + tin	g.
Wt. of oxygen	<hr/> g.
Wt. of crucible + oxide of tin	g.
Wt. of crucible, empty	g.
Wt. of oxide of tin	<hr/> g.

b. Calculate the percentage composition of the oxide of tin.

c. Find the atomic weight of tin in the table and calculate the formula of the oxide.

d. From the data obtained in Ex. 13, calculate the formula of the hydrate. In 13 a, the formula is $y\text{CaSO}_4 \cdot x\text{H}_2\text{O}$.

Wt. of water \div Formula wt. of water ($\text{H}_2\text{O} = 2 \times 1.008 + 16 = 18.016$) = x .

Wt. of residue \div Formula wt. of residue ($\text{CaSO}_4 = 40 + 32 + 4 \times 16 = 136$) = y .

$$\frac{\text{wt. of water}}{\text{wt. of calcium sulphate}} = \frac{18.016 \times x}{136 \times y} = \frac{x(\text{H}_2\text{O})}{y(\text{CaSO}_4)}$$

Divide both x and y by y . This will give the smallest whole

numbers which are in the same ratio. Then substitute these whole numbers for x and y in $y\text{CaSO}_4 \cdot x\text{H}_2\text{O}$.

In 13 b, the formula is $y\text{BaCl}_2 \cdot x\text{H}_2\text{O}$, and the values of x and y are found in the same way.

EXERCISE 18.

SOLVENTS AND SOLUBILITY.

(With practical applications.)

Object: *To find out how to hasten the process of solution, and the difference between solution and suspension. To learn what solvents dissolve certain common materials.*

Apparatus: Test-tubes and rack. Corks to fit. Mortar. Graduated cylinder. Glass rod.

Materials: Cupric sulphate (hydrate). Filter paper. Potassium permanganate. Powdered rosin. Alcohol (95% denatured). Fat (or lard). Carbon tetrachloride. Paraffin. Gasoline (or benzene).

a.* A mass dissolves at its surface only. The larger the total surface, the faster it dissolves.

Take two large crystals of cupric sulphate of equal size and yet of such size that each can be slipped into a test-tube. Provide two dry test-tubes with corks to fit. Pulverize one of the crystals very finely in the mortar, so that no large particles are visible. Place the powder in one test-tube and the crystal in the other. Add from the graduated cylinder 20 c.c. of water to each. Cork the tubes quickly, note the time on a watch(?), and shake the tubes gently. When the powder has all dissolved, note the time again(?). Estimate the fraction of the original crystal which remains in the other tube, then continue shaking it until it also has dissolved, and note the time again(?). Compare the total times required to dissolve each(?). Why does pulverization make this differ-

* One pupil can do a while his neighbor does b, each observing the other's results.

ence in the rate of solution? To save time, in what form should you use a substance, to be dissolved?

b. Take again two nearly equal-sized, but **smaller crystals** of cupric sulphate. Fill two test-tubes with water. Put one of the crystals into one of the tubes. Drop the other crystal into the second tube (Fig. 12), so that it is covered by the water, but rests on a narrow strip of ordinary paper. Set both tubes upright in the rack. How long does each crystal take to dissolve?

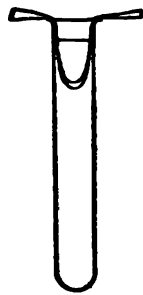


FIG. 12

If you had to dissolve a large amount of material quickly, with the least expenditure of effort, how should you proceed (in answering, take the results of both a and b into account)? While the tubes are standing, go on with c, d and e.

c. On to a strip of paper dipping under water (Fig. 12), drop a crystal of potassium permanganate(?). Explain(?).

d. Place 1 c.c. of powdered rosin in each of two dry test-tubes. Add to one 5 c.c. of alcohol and to the other 5 c.c. of water and shake both(?).

Pour the alcoholic liquid into a large beaker full of water. In what ways is a suspension like a solution(?) and how does it differ?

Should you remove rosin (or varnish or a similar gum) from clothing with water? With alcohol?

e. Place a small piece of lard, half the size of a pea, in each of two test-tubes. Add 2 c.c. of water to one and 2 c.c. of carbon tetrachloride* (or carbon disulphide or ether) to the other and shake(?). How should you remove grease from clothing? (In removing a grease-spot, place the part flat on a

* Non-inflammable. Carbon disulphide or ether must not be used near a flame.

piece of blotting paper, to absorb the solution, and rub the spot with a rag dipped in the solvent.)

f. Use small pieces of solid paraffin as in e, employing water and gasoline or benzene as solvents.

g. Take 10 c.c. of water in each of three test-tubes. To one add a single drop of alcohol and shake. Does the alcohol dissolve? Add more alcohol, a few drops at a time, until about 5 c.c. have been added. Has the amount added yet become greater than the water can dissolve?

To the second tube add carbon tetrachloride (or carbon disulphide) in the same way, and answer the same questions(?).

To the third add gasoline (or benzene) in the same way, and answer the same questions.

If you had a mixture of sugar (which is soluble in water) with fat on a piece of cloth, how could you remove first one and then the other component of the mixture?

EXERCISE 19.

SOLUBILITY AND TEMPERATURE. SATURATION.

Object: *To learn how to make a saturated solution, how to know approximate amount dissolved (approx. solubility). Also to study the influence of temperature on solubility.*

Apparatus: Test-tubes. Funnel. Watch glass. 100 c.c. beaker. Trip scales.

Materials: Calcium sulphate (pulv.). Calcium carbonate (pulv.). Filter-paper. Potassium dichromate (cryst.).

a. **Test of Degree of Solubility** (two pupils working together). Place 1 c.c. of powdered calcium sulphate in a test-tube and shake with 10 c.c. of water for two or three minutes. Filter the mixture, catching the clear filtrate in a clean test-tube. Treat as in Ex. 11 a, to see whether any dissolved(?). The second pupil uses 1 c.c. of powdered calcium carbonate (chalk) in the same way(?). Compare the deposits on the watch glasses(?). Which substance is more soluble? What

conclusion should you have drawn from mere shaking with water, without completing the test?

Was the solution you evaporated saturated? Was it concentrated? What is a saturated solution?

b. Pulverize 6 g. of potassium dichromate. Describe the change in color and explain it(?). Shake the powder with 10 c.c. of water in a test-tube until the liquid is saturated. What evidence is there that a good deal dissolves?

Now warm the contents of the test-tube gently, with occasional shaking(?). Is the substance more or less soluble as the temperature rises? When all has dissolved, set the tube in the rack and examine it when it has cooled(?). Describe the contents.

Warm the contents of the tube carefully (to avoid cracking the tube) once more until all has dissolved. Then hold the tube in running water to cool it rapidly(?). Describe the contents(?). Note two differences (in size of particles and in color) between the results of slow and of rapid cooling and explain each(?).

EXERCISE 20.

HYDROGEN CHLORIDE—PREPARATION AND PROPERTIES.

Object: *To obtain hydrogen chloride and to learn some of its properties.*

Apparatus: Test-tubes. Glass rod. Watch glass. Lens (one for class).

Materials: Ammonium chloride. Calcium chloride. Potassium chloride. Sulphuric acid (conc.). Litmus paper. Ammonium hydroxide (sol.). Sodium chloride. Silver nitrate (sol.). Zinc dust. Sodium-hydrogen sulphate (sat. sol.).

a. Place 1 c.c. of any chloride (for example ammonium chloride NH_4Cl , calcium chloride CaCl_2 , or potassium chloride KCl) in a test-tube and add 1 c.c. of concentrated sulphuric acid(?). Is the material boiling (feel the bottom of the

tube. Sulphuric acid boils above 300°)? Waft a little of the gas towards the nose, but do not bring the latter too near to the tube (odor?).

To learn the behavior of the gas with water vapor, blow the breath across the mouth of the tube(?).

Moisten pieces of blue and of red litmus paper with water (to dissolve the gas) and place them in the mouth of the test-tube(?). What chemical property does the result show the aqueous solution of the gas to possess?

Dip a glass rod in ammonium hydroxide solution. Smell the rod(?). The gas which is dissolved in, and given off by this solution is ammonia (NH_3). Now insert the glass rod into the mouth of the test-tube(?). The product is formed from the ammonia gas and the hydrogen chloride gas.

Light a wooden splint and plunge the flame into the test-tube(?). Does the gas burn or support combustion?

Four properties of hydrogen chloride (or its solution) have been observed. Make a list of these, and note opposite each whether it is a physical or a chemical property [41].

b. Of what elements is hydrogen chloride composed? What is its formula? What proportions by weight of the constituents are indicated by this formula?

Write the equation for the original action by which, in a, you obtained the gas. Note that the three chlorides suggested will give, respectively, $(\text{NH}_4)\text{HSO}_4$, CaSO_4 , and KHSO_4 , as one of the products. Where is this one of the products? To which of the varieties of chemical change does this action belong [146]?

c. Write the equation for the interaction of ammonia and hydrogen chloride. To which of the five varieties of chemical change [147] does this action appear to belong?

d. Take 1 c.c. of sodium chloride in a dry test-tube and add 1 c.c. of concentrated sulphuric acid [139]?

Place in a second test-tube 0.5 c.c. of silver nitrate solu-

7001. Dip a clean glass rod in water and hold the rod, with the adhering water in the hydrogen chloride, **first test-tube** for one minute. Then transfer the rod, with the **adhering solution** to the second test-tube and mix the liquids (?).

To obtain a better idea of the action, add to the **same silver nitrate solution** one drop of concentrated hydrochloric acid (?). This precipitate is described as "curdy."

e. Place on the watch glass a few particles of **zinc dust**. Dip a glass rod in water, hold it in the hydrogen chloride as in d, and then place the drop of the aqueous solution of the gas on the zinc dust (?). What is the gas liberated (See Ex. 9 c.)? Make the equation for this action ?.

Name three other metals which would react in a **similar way** with hydrogen chloride solution? Write equations for these three actions ?.

f.* Make the equation for the action in par. 1 of d, and write the name of the substance under each formula(?).

Take 5 c.c. of saturated sodium-hydrogen sulphate solution. Add to it concentrated hydrochloric acid, a **very little** at a time, shaking the **mixture** after each addition(?). Examine the precipitate with a lens and describe the form of the particles(?). Make the equation for this action(?).

Compare the two equations last written and read §83. What substance, by its escape as a gas from the mixture, **permitted** the first action to go to completion? If this substance had remained dissolved, what difference would this have **made** in the result? What substance, by its separation as a **precipitate**, permitted the second action to go to completion? **Explain** why the separation of a product permits the completion, of an action(?).

* This experiment very effective in the class-room also.

EXERCISE 21.

CHLORINE—PREPARATION AND PROPERTIES.

Object: *To prepare chlorine and to observe some of its properties.*

Apparatus: Trough. Test-tube and cork to fit. Kipp's hydrogen generator (one for class). Taper.

Materials: Potassium permanganate. Hydrochloric acid (conc.) Ammonium hydroxide (sol.).

Caution: Chlorine gas, when breathed, has a very irritating action in the throat. Breathing the vapor of alcohol (sprinkled on a handkerchief) or ammonia gives relief. Perform the following experiments in a HOOD, or place where there is a good draft.

a. Fill the trough with water. Place in the test-tube a very few crystals of potassium permanganate and add cautiously a drop or two of concentrated hydrochloric acid (make sure that you do NOT add some other acid by mistake). Leave the tube open (uncorked). What is the color of the gas?

As soon as the tube appears to be filled with the gas, close the tube quickly with the cork, invert the tube instantly in the water contained in the pan and pull out the cork. Keeping the mouth under the water, shake the tube and note whether or not the gas dissolves (?).

b. Place about 1 c.c. of potassium permanganate in the 300 c.c. beaker. Add 2-3 c.c. of concentrated hydrochloric acid and cover with a glass plate.

When the beaker is filled with the gas, move the plate aside and insert for a few seconds a jet of hydrogen burning at a nozzle attached to a Kipp's apparatus* (?). Keep the beaker covered as far as possible. While the hydrogen burns in it, is there any change in the color of the gas? What gas was formed? Dip a glass rod in ammonium hydroxide solution and

* If no Kipp is available, a single generating bottle may be used for the whole class, or each pupil may take zinc and hydrochloric acid in a test-tube provided with a stopper, L-tube, rubber connection, and nozzle.

insert it into the gas left in the beaker (?). Cover the beaker again and use for c.

c. When the beaker from b has again become filled with chlorine, light a taper and plunge the flame into the gas (?). The wax of the taper is composed chiefly of compounds of carbon and hydrogen. The black product is carbon(soot). What compound must have been formed? Verify your conclusion by testing the gas with ammonium hydroxide on a rod (?).

As soon as the last experiment is finished, take the beaker (covered) to the sink, pour some ammonium hydroxide solution into it. Cover the beaker again with the plate and shake, holding the whole over the sink, and finally wash out.

d. What did you observe that showed chlorine not to be lighter than air? What two other physical properties and two chemical properties were observed?

EXERCISE 22.

SODIUM HYDROXIDE.

Object: *To learn some of the properties of bases.*

Apparatus: Test-tubes. Glass rod.

Materials: Sodium hydroxide(sol.). Litmus papers. Cupric sulphate(sol.). Aluminium sulphate(sol.). Phenolphthalein(sol.). Ammonium hydroxide(sol.).

a. Recall the action of sodium on water [58], as seen in the class-room. Write the equation for this action, and place the name of the substance under each formula(?). To which of the five varieties of chemical change [147] does this action belong?

b. Take 1 c.c. of sodium hydroxide solution and dilute it with 10 c.c. of water. Dip a clean glass rod in the liquid and taste it [Immediately rinse the mouth out with water. Taste substances only when told to do so](?). Dip red and

blue litmus paper in the solution(?). Rub the liquid between the fingers(?). Use the same solution in c.

c. To 1 c.c. of cupric sulphate solution add one-third of the diluted sodium hydroxide solution(?). Make the equation and attach to each formula the name of the substance(?). To which variety of chemical change [147] does this reaction belong? Where is the other product of the reaction and how should you proceed so as to separate the precipitate, and finally obtain the other product in solid form?

d. To 1 c.c. of aluminium sulphate solution add 1 or 2 drops (shake between drops) of the sodium hydroxide solution(?). Make the equation and answer the same questions(?).

e. To the rest of the sodium hydroxide solution add one drop of phenolphthalein solution(?). Litmus and phenolphthalein are called indicators. How does each behave with a base?

f. What substance have we previously found to have the same effect on phenolphthalein as has sodium hydroxide(Ex. 11)? What is its formula?

Dilute 1 c.c. of ammonium hydroxide solution with 10 c.c. of water and try with it the tests in b and d(?).

g. Make a list of the chemical properties you have found a base to possess(?). What radical is common to all bases, and confers these properties?

EXERCISE 23.

PROPERTIES OF ACIDS—IONIZATION.

Object: *To learn about ionization and the properties of acids.*

Apparatus: Test-tubes. Glass rod.

Materials: Sodium hydroxide. Hydrochloric acid(dil.). Litmus papers. Sodium hydroxide(sol.). Hydrochloric acid(conc.).

a. Place a small piece of sodium hydroxide in a test-tube, add 2 c.c. of water, and shake(?). What property does the

solution possess which the solid body lacked [202, par. 3]? According to the theory of ionization, what chemical change took place when the solid dissolved [204]? Make an ionic equation for this change [209].

What evidence, based on the freezing and boiling-points of such a solution, shows that this change has occurred? How many new substances are produced in this action? Name them(?).

b. You dissolved hydrogen chloride in a drop of water (Ex. 20 d). Answer the same five questions as in a, and make the ionic equation.

In the case of every other simple acid, base, or salt, would the answers always be the same, or would they in some cases be different?

c. Take 2 c.c. of dilute hydrochloric acid and dilute it with 10 c.c. of water. Repeat with this solution the three tests described in Ex. 22 b, and record the results.

d. When zinc (or one of certain other metals, Ex. 9 d) is placed in dilute sulphuric acid, what gas is liberated? Make the ionic equation for this action [212].

All soluble acids show the properties found in c and d. Make a list of these properties(?).

e. Place 3 c.c. of sodium hydroxide solution in a test-tube. Add about 1 c.c. of concentrated hydrochloric acid. Is there evidence of chemical action (touch the back of the hand with the bottom of the test-tube)? Keep the mixture for f.

Make the ordinary [202] and ionic [211] equations for this action, which is a "neutralization."

f. Since acids and bases act oppositely on indicators, like litmus and phenolphthalein, it is possible to find out when the proportions of the two materials required for complete inter-action have been taken, for the mixture will then be without action on an indicator.

Dilute the mixture prepared in e. Moisten a glass rod in

it, and touch the edge of a piece of blue and of a piece of red litmus paper with it(?). If the blue turns red, there is excess of acid. Add sodium hydroxide solution a drop at a time, shaking between drops, and moistening the rod and testing as before. Continue until no effect is produced on litmus of either color. If, by accident, too much alkali is added, or if the mixture was alkaline in the beginning, use drops of dilute hydrochloric acid in the same way.

When the liquid is neutral to indicators, taste it by touching the tongue with the glass rod(?).

Set the solution aside to evaporate by itself. When it has dried up, examine the solid. What is the form of the crystals? What is the substance?

EXERCISE 24.

TITRATION OF BASES AND OF ACIDS.

Object: *To learn how to measure the quantity of an acid or of a base contained in a given specimen (quantitative).*

Apparatus: Burette. Beaker. Glass rod (thin).

Materials: Normal acid.* Sodium hydroxide (sol.). Phenolphthalein (sol.). Vinegar (white). Litmus (sol.).

a. A normal solution of any acid is one containing 1 g. of the hydrogen radical(H) per liter, accompanied, of course, by an equivalent amount of the negative radical of that acid. Thus, one liter of normal hydrochloric acid(HCl) will contain $1 + 35.46$ g. of the acid. Similarly, a normal solution of a base contains 17 g. of hydroxyl radical(OH) per liter. Thus one liter of normal sodium hydroxide(NaOH) will contain $23 + 17$ g. of the base. What weight of acetic acid (H)(CO₂CH₃) is contained in one liter of the normal acid? What weight of potassium hydroxide in one liter of the normal solution of this base?

* 63 g. oxalic acid H₂C₂O₄ · 2H₂O per liter.

b. **Titration of a base with a standard acid.** Fill a burette with the normal acid. Allow some of the acid to flow out, until all air has been expelled from the tip, and until the meniscus has reached the graduated portion of the burette. Hold the burette so that the surface of the acid is on the same level with the eye, and read the level of the bottom of the meniscus (Fig. 3, Ex. 2 a). Observe that the graduation is downwards, so that a reading two-tenths of a c.c. above the 2 c.c. mark is 1.8 c.c. (not 2.2 c.c.). Record the reading (?).

Take exactly 10 c.c. of sodium hydroxide solution in the graduated cylinder (dry this first). Pour it into a beaker. Rinse out the cylinder three times with a little water (about 5 c.c. each time). Set the beaker on a sheet of white paper under the burette, and add a drop or two of phenolphthalein solution (?). Stir vigorously with a very thin glass rod.

Now allow the acid to run in a rapid succession of drops into the beaker. Stir vigorously after each addition of a few drops. As soon as the acid begins to decolorize the indicator round the point where it enters, proceed more cautiously. Add only one drop at a time, and stir. The aim is to have the solution distinctly pink before the final drop of acid is added, and perfectly colorless when that drop has been allowed to flow in. If, at the first attempt, you over-shoot the mark, wash out the beaker, and take a fresh portion of sodium hydroxide solution, read the level of the acid in the burette, and try again.

When the titration has been successfully performed, read the level of the acid in the burette (?). Subtract the reading from that made at the beginning (?). The difference (?) is the volume of normal acid required for complete interaction with (neutralization of) 10 c.c. of the solution of the base.

c. Since one liter of the normal acid contains 1 g. of available hydrogen, 1 c.c. of the acid contains 0.001 g. of hydrogen

radical. Calculate the weight of hydrogen radical used in your experiment(?).

Now, 1 g. of hydrogen radical interacts with 17 g. of hydroxyl(OH). Using the weight of hydrogen just found, calculate the weight of hydroxyl contained in the portion of base you took(?).

Finally, using the proportion indicated in the formula NaOH, calculate the total weight of sodium hydroxide which contains the weight of hydroxyl you found(?).

d. Knowing from the result of c the weight of sodium hydroxide in 10 c.c. of the solution, calculate the weight per liter(?).

Calculate the concentration of the solution in terms of a normal solution of sodium hydroxide as unity(?).

e. **Titration of an acid.** Wash out the burette, fill it with the sodium hydroxide solution as in b, and read the level of the meniscus(?).

In c, par. 2, we found the weight of hydroxyl in 10 c.c. of this solution. Calculate the weight in 1 c.c.(?). We may now use this solution containing a known concentration of a base for measuring quantities of acids.

f. Take exactly 10 c.c. of white vinegar in the graduated cylinder(dry this first). Pour it into a beaker, rinse the cylinder three times with water, and set the beaker on a piece of white paper under the burette. Add 4 drops of litmus solution, or enough to give the liquid a distinct, but not strong pink color. Stir with a thin glass rod.

Now titrate this solution with that in the burette, exactly as described in b(third par.). In this instance the color appearing locally will be blue. Aim to stop when the tint is pinkish-violet, half way between pink and blue. Repeat, if necessary to secure a sharp result. Then read the level of the meniscus(?) and subtract from the former reading to learn the volume of base used(?).

g. Using the weight of hydroxyl per c.c. found in **e** (second par.), calculate the weight of hydroxyl used in **f** (?).

This weight will neutralize $\frac{1}{17}$ as much hydrogen radical (in the acetic acid contained in the vinegar). Calculate the weight of acid hydrogen in the 10 c.c. of vinegar (?).

Finally, using the formula of acetic acid $(H)(CO_2CH_3)$, calculate the total weight of acetic acid in the 10 c.c. of vinegar (?). How much is this per liter? What per cent?

These methods, and others like them, are known as **volumetric methods**, and are largely used in analyses made for commercial (as well as scientific) purposes.

EXERCISE 25.

VALENCE OF A METAL BY DISPLACEMENT.

Object: *To determine; (1) the weight of hydrogen which is displaced by one at. wt. (24.3) of magnesium,* and (2) the valence of magnesium. If, e.g., 24.3 g. of magnesium is found to displace (and combine in place of) one at. wt. (1.008 g.) of hydrogen, the atomic weight of magnesium is univalent. If it displaces two at. wts. of hydrogen (2.016 g.), it is bivalent, and so forth (quantitative).*

FIRST METHOD.

Apparatus: Balance. 200 c.c. Flask. Stopper (1-hole). L-tube and rubber delivery tube. 2-liter (or 1-liter) bottle and glass plate or cork. Trough. 50 c.c. graduated cylinder. Funnel. Thermometer. Barometer. 500 c.c. graduated cylinder (1 for 10 pupils).

Materials: Magnesium wire (2 mm. diam.), or iron piano-wire, or aluminium wire. Hydrochloric acid (pure, conc.).

a. Take a piece of magnesium † wire weighing about 1.7 g. and determine (balance—if none free at the moment, proceed

* With another metal the same directions and questions apply, provided only the name of the metal be substituted for that of magnesium throughout, and the appropriate atomic weight (iron, 55.8; aluminium, 27.1) is employed in the statement of the object and in **d** and **e**. If one method only is used, try the third.

† Other metals, such as aluminium (wire, about 1.4 g.), may be used.

with next par.) its exact weight to the nearest centigram(?). Tabulate the data as shown under d.

Fit the 200 c.c. flask with a one-hole stopper, L-tube, and rubber delivery tube, and test for air-tightness(Ex. 7 d). Fill the 2-liter bottle* with water and invert it on the support in the trough. Place in the 500 c.c. graduated cylinder 20 c.c. of concentrated hydrochloric acid and dilute to 200 c.c. with water. Set the funnel in the flask, and pour into it the acid from the graduated cylinder. In removing the funnel, be careful not to wet the neck of the flask with the acid.

Coil up the magnesium wire so that it can be pushed into the neck of the flask, when the latter is held in an inclined position. The coil must be so large that the wire sticks in the neck of the flask and does not slide into the acid. Replace the stopper in the flask and insert the delivery tube into the mouth of the 2-liter bottle. Now, shake the flask slightly so as to bring the acid in contact from time to time with the metal. When the wire falls into the acid, place the body of the flask immediately in the water in the trough. This will tend to keep the acid cool, and prevent the heat of the reaction from raising the temperature of the acid and causing the action to proceed too rapidly. When the magnesium has all disappeared, remove the delivery tube.

b. Lower the bottle in the trough(adding water, if necessary) until, on looking horizontally, with the eye at the level of the water, the levels inside and outside are seen to be the same. Incline the bottle, if necessary, to accomplish this. While the bottle is in this position, cover the mouth with a glass plate(or insert a cork), and set the bottle with its contents erect upon the table.

* A 1-liter bottle(or 1-l. graduated cylinder) if available is better, because about 0.9 g. of magnesium(or 1.8 g. of iron, piano-wire, or 0.7 g. of aluminium wire) and 8 c.c. of the acid diluted to 100 c.c. are then used and time is saved.

Read the temperature of the water(?). Ascertain also the height of the barometer(?).

If there is not time to proceed with the operations directed below, the bottle and its contents may be set aside until the following period.

c. The weight of this hydrogen is to be found, not by weighing it ourselves, but by measuring its volume, and using the fact that 1 liter of the dry hydrogen at 0° and 760 mm. weighs 0.09 g.(see d).

d. Use a graduated cylinder(500 c.c.) to fill the bottle once more to the top with water, noting carefully the volume of water required(?).

The hydrogen was mixed with water vapor. Find the tension of the aqueous vapor at the temperature recorded in a [p. 423](?) and subtract this from the barometric height [54](?).

Reduce the volume of hydrogen, from the observed temperature and barometric pressure as just corrected, to 0° and 760 mm. [51, 52, 53](?).

Calculate the weight of this hydrogen(1 liter weighs 0.09 g.)?

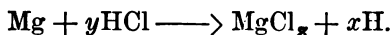
Weight of metal taken	g.
Volume of hydrogen obtained	c.c.
Temperature	°
Barometric height	mm.
Tension of aqueous vapor	mm.
Pressure of hydrogen, corrected	mm.
Volume of hydrogen at 0° and 760 mm.	c.c.
Weight of this hydrogen	g.

e. The atomic weight of magnesium in 24.3. State just what experimental fact this sentence represents [92](?).

f. The weight of hydrogen found in d was that displaced by the weight of magnesium taken in a. Calculate from these data the weight of hydrogen which would be displaced by one

atomic weight(24.3 g.) of magnesium(?). What is the nearest whole number of atomic weights of hydrogen(1.008 g.) contained in this weight of hydrogen? This whole number is the valence of magnesium [222]. Mg therefore displaces xH . Supply the value you have found for x (?).

g. How many formula weights of hydrochloric acid(HCl) are required to furnish this amount of hydrogen? Now make the complete equation, supplying the values you have found for x , y , and z :



You did not measure the weight of Cl_z , or determine the value of z . What law of chemistry enables you to supply this value without measurement [35]?

h. If hydrogen is univalent, what is the valence of oxygen in water(H_2O)[225]? Write the formula of magnesium oxide(?).

j. If the formula of hydrogen sulphide is H_2S , what is the formula of magnesium sulphide?

SECOND METHOD.

Apparatus: Wide test-tube with 2-hole stopper to fit. Funnel. Pinch-clamp, rubber connection and glass tubing. Rubber delivery tube. Iron stand, ring, and clamp. 1-liter bottle. Trough. Thermometer. Barometer. 500 c.c. graduated cylinder(1 to 10 pupils).

Materials: Magnesium ribbon, or iron piano-wire, or aluminium wire, or zinc(C.P. gran.). Hydrochloric acid(pure, conc.).

a. Take a piece of magnesium ribbon weighing about 0.9 g.* and determine(balance—if all are occupied, proceed with the next par.) its exact weight to the nearest centigram(?). Tabulate all data as shown under d(First Method).

* For a 2-liter bottle, take 1.8 g. of magnesium. For other metals (and 1-liter bottle) use iron(piano-wire, about 0.9 g.), aluminium(wire, about 0.7 g.), or zinc(pure, granulated, about 1 g.). If zinc is used, weigh the test-tube first empty and again with the zinc, getting the weight of the latter by difference: also coil the platinum wire and place it amongst the zinc(the glass rod need not be detached).

Fit a wide test-tube with a 2-hole stopper, straight tube reaching almost to the bottom, funnel, pinch-clamp, and short rubber connection. Set this up as in Fig. 13, using the triangle on a ring to support the funnel, and a clamp to hold the test-tube. The L-tube must not project below the bottom of the stopper. Test for air-tightness(Ex. 7 d).

Coil up the magnesium ribbon, place it in the test-tube, and replace the stopper. Through the funnel, fill the apparatus completely, from the tip of the delivery tube to the clamp below the funnel, with water. Close the clamp when the funnel has almost emptied itself. Invert the 1-liter bottle(or a 1-liter graduated cylinder), filled with water, on the support in the trough, and insert the delivery tube in the mouth of the bottle.

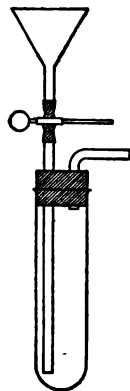


FIG. 13

Fill the funnel with pure, concentrated hydrochloric acid, and admit this to the test-tube, a little at a time, in such a way that a steady, rapid, but not violent action takes place. Replenish the funnel, if necessary, so that the funnel never becomes entirely empty. A good deal of the acid may be required at first(especially with other metals than magnesium), before sufficiently rapid action sets in. When the metal has all disappeared, drive all the gas over into the bottle by pouring water once more through the funnel(be careful that no air is carried in with the water).

b to j. Follow the directions given under the First Method.

THIRD METHOD.

(Easily applicable to magnesium only.)

Apparatus: Balance. 250 c.c. bottle. Trough(stone ware or enamelled). 50 c.c. graduated cylinder. Glass plate. Thermometer. Barometer

Materials: Magnesium ribbon. Hydrochloric acid(conc.).

a. Weigh(balance) to the nearest centigram exactly 2 meters of magnesium ribbon(?). Calculate what length will weigh exactly 0.2 g. and cut off this length for use. Record all data as shown under d(First Method). Roll the ribbon into a spiral coil somewhat smaller than the inside diameter of the mouth of the 250 c.c. bottle.

Place in the trough about 5 cm. depth of water. Measure into the bottle 30 c.c. of hydrochloric acid(conc.) and fill it up to the brim with water. Cover it with a glass plate and invert it in the trough, leaving the glass plate in the trough with the bottle inverted upon it. If any air gets into the bottle, lift out the bottle with the plate firmly pressed against the mouth, fill it up with water and try again.

Place the spiral of magnesium ribbon in the trough and slip the mouth of the bottle over it, setting the bottle down firmly on the bottom of the trough. When the magnesium has all disappeared, slide the bottle back on to the glass plate.

b to j. Follow the directions given under the First Method.

EXERCISE 26.*

SALTS.

Object: *To prepare lists of the three ways of liberating elements and the six ways of preparing salts which have been met with during the preceding work.*

Apparatus: Test-tubes. Trip scales. Watch glass. Beaker.

Materials: Magnesium ribbon. Sulphuric acid(dil.). Sodium carbonate. Calcium chloride.

a. Put 5 cm. of magnesium ribbon in dilute sulphuric acid in a test-tube(?). What is the gas given off? What other

* Exercises 26 and 27 are placed here as part of the review following Chap. XV. If more convenient, they may precede Ex. 25, and form part of the work accompanying Chap. XIV.

product is formed and how should you obtain it in solid form? Write the equation, assuming Mg to be bivalent(?). Write an ionic equation also(?). Which of the varieties of chemical change is here illustrated?

Give two other ways of liberating an element, with one illustration of each(?).

b. Place 2 g. of sodium carbonate in one test-tube and 1 g. of calcium chloride in another. Dissolve each salt in 10 c.c. of water. Heat the calcium chloride solution to boiling and add to it a little sodium carbonate solution(?). Shake, wait until the precipitate has settled, and add one more drop of the sodium carbonate solution. Continue the three operations described in the last sentence until the last drop produces no further precipitation in the clear part of the liquid. Then filter the mixture and evaporate two or three drops of the clear filtrate in a watch glass as in Ex. 10 f(?).

Write the formulæ of the two salts taken(?). Separate the radicals in brackets thus $(\text{NH}_4)(\text{NO}_3)$. Complete the equation as a double decomposition(?). Write the names under the formulæ of the products(?). Write the equation in ionic form also(?).

Define each of the two ways of forming salts which are illustrated in b(?). Define a third way used in Ex. 23 ef. What have these three ways in common? Define a fourth way met with in Ex. 26 a(?). Define a fifth way which was illustrated in Ex. 5 d, Ex. 16 and §§23 and 167(?). Define a sixth way illustrated in Ex. 6 c.

EXERCISE 27.

INCOMPLETE REACTIONS.

Object: *To study a reversible action, to show that it is incomplete, and to try one of the ways of carrying such a reaction to completion*
[Read 83, also 163, 207].

Apparatus:, Trip scales. Test-tubes. Funnel. Glass rod.

Materials: Calcium chloride(fused or gran.). Oxalic acid. Filter paper. Labels. Hydrochloric acid(conc.). Ammonium hydroxide(sol.).

a. Weigh out(trip scales) 1 g. of calcium chloride(fused or gran.) and 1.2 g. of oxalic acid. Dissolve each, separately, in 10 c.c. of water, and heat the solutions to the boiling point. Add the oxalic acid solution to the calcium chloride solution, a little at a time, boiling for two minutes and allowing the precipitate to settle between the additions. When one drop ceases to produce any fresh precipitation in the clear part of the liquid, note what proportion of the original oxalic acid solution remains unused(?). Add this remainder, thus making sure that there is an excess of oxalic acid present, beyond that apparently necessary to precipitate all the calcium.

The radicals are $(\text{Ca})(\text{Cl})_2$ and $(\text{H})_2(\text{C}_2\text{O}_4)$. Write the equation for the action(?). Write the name under the formula of each substance(?), and a downward arrow beside that of the precipitate(?). To what variety of chemical change does this one belong?

b. Proceed now to find out whether this reaction is incomplete or practically complete. To do this, shake the mixture and pour it all on to a filter(Fig. 5, Ex. 5). Catch the filtrate in a clean test-tube, label it *F*(use in c). Set a vessel under the funnel, and wash the precipitate(calcium oxalate) with water. To do this, pour water, a little at a time, from a test-tube on to the filter, so as to wash every part of it. When the filter has drained, set a test-tube under the funnel, puncture the filter-paper with a glass rod, wash the calcium oxalate through into the test-tube.

If the reaction was reversible, the equation, when read backwards, represents a possible chemical action. Name the interacting substances in the reversed action(?). To the calcium oxalate, suspended in water, add the required reagent(?).

Was the action a reversible one? The conclusion may be confirmed as follows:

c. If this reverse action occurs, it was in operation in the original mixture made in a. In that case, all the calcium could not have been precipitated, some must be present in the filtrate *F*, and the reaction was incomplete. The reaction may be completed by removing (or chemically altering) one product. Destroy the hydrochloric acid by adding to the filtrate *F* ammonium hydroxide a drop at a time and shaking (?). What does the result prove?

EXERCISE 28.

GRAPHITE.

Object: *To try the action of solvents, and of solutions of acids and bases on graphite, to see whether it dissolves in the former, or interacts with the latter.*

Apparatus: Test-tubes.

Materials: Graphite (pulv.). Hydrochloric acid (dil.). Ammonium hydroxide (sol.). Carbon tetrachloride or disulphide.

a. Place not over 0.2 c.c. of graphite in each of three test-tubes. Add 5 c.c. dilute hydrochloric acid, as an example of an acid, to one. Add 5 c.c. of ammonium hydroxide, chosen as a base, to the second. Add 4 c.c. of carbon tetrachloride, chosen as a solvent, to the third.

Shake the last vigorously (away from all flames). Does the graphite dissolve? Name a solvent for carbon (?).

b. Boil the two other samples with the acid and base, respectively. Is there any evidence of continued action, when the tube is taken out of the flame? Conclusion (?).

What are the uses of graphite?

EXERCISE 29.

CARBON DIOXIDE BY OXIDATION.

Object: *To observe the production of carbon dioxide when wood burns and oxides of metals are reduced by carbon.*

Apparatus: 250 c.c. bottle. Mortar. Trip scales. Hard glass test-tube with 1-hole stopper and L-tube.

Materials: Limewater. Splints. Cupric oxide (pulv.). Charcoal (pulv.). Stannic oxide (pulv.). Lead monoxide.

a. Place 10 c.c. of limewater in a 250 c.c. bottle. Burn a splinter of wood in the bottle, taking care not to drop any ashes into the liquid. Close the bottle with the hand and shake(?). If the change is not distinct, repeat the burning, using the same limewater, and shake again. What elements does wood contain [258]? Make the equations for the reactions of the carbon dioxide with the limewater [252].

b.* Mix intimately in the mortar 5 g. of cupric oxide and 0.5 g. of powdered charcoal. Place this on a strip of paper, folded V-shape, and slip the hard-glass test-tube over the paper. Turn the tube round, so as to leave the mixture in it, and withdraw the paper. Insert the stopper and L-tube. Place 10 c.c. of limewater in a test-tube. Clamp the tube containing the mixture in a horizontal position, at such a height that it can be heated, and let the L-tube project to the bottom of the limewater. If the tube is too short to reach the bottom, add a straight glass tube and rubber connection. The purpose is to compel any gas which may be given off to bubble through the entire depth of the limewater.

Heat the mixture, beginning at the end farthest from the stopper, at first gently and then strongly and persistently. What change takes place in the limewater? Finally, take the tube out of the limewater and then remove the flame. When

* b, c and d may be done by different pupils and the results compared.

the tube is cold, grind the contents in the mortar with water, and wash away the lighter particles. Examine the residue(?). Make the equation for the action [252](?). What important industrial processes depend on this reaction? What was oxidized and what reduced in this reaction?

c. Perform the same experiment, using 5 g. of oxide of tin and 1 g. of charcoal. Answer the same questions as in a.

d. Perform the same experiment, using 2 g. of litharge (lead monoxide) and 2 g. charcoal. In this instance heat gently. Answer the same questions as in a.

EXERCISE 30.

CARBON DIOXIDE—PREPARATION AND PROPERTIES.

Object: *To prepare carbon dioxide and to observe its properties.*

Apparatus: Gas generating bottle (90 c.c.), 2-hole stopper, thistle tube, L-tube and rubber delivery tube. 3 w.-m. bottles (250 c.c.) and glass plates. Test-tubes. Taper.

Materials: Marble (chips). Hydrochloric acid (dil.). Litmus (sol.). Limewater.

a. Use the apparatus in Fig. 10, Ex. 10 a. Slide into the bottle (do not drop them in) enough marble chips to fill it to a depth of one inch, and connect. Add dilute hydrochloric acid and fill all bottles with the gas by upward displacement of air. Cover the bottles with glass plates.

To a test-tube (clean) full of water add some litmus solution. Pour half of the mixture into another test-tube and keep the latter for reference. Through a clean glass tube, pass carbon dioxide into the other half (see c).

b. Light a taper and pour the gas from one of the bottles over the flame, as you would water(?). Does the gas support combustion? Is the gas heavier or lighter than air? Give the reason for your answer(?).

To learn whether the gas is soluble in water, pour about

10 c.c. of water into a bottle of the gas, cover quickly and firmly with the palm of the hand, and shake vigorously. Is the hand held against the mouth of the bottle by atmospheric pressure, or not? Is the gas soluble?

To ascertain whether the gas diffuses upwards, set the emptied bottle(now filled with air) over the full one, and mouth to mouth with it, and withdraw the plate. After ten minutes(meanwhile, do c), replace the glass plate between the bottles, remove the upper bottle, along with the plate, and set it upright upon the table. Slip the plate aside for a moment, add some limewater, replace the plate, and shake(?). Did the greater density of the carbon dioxide prevent its diffusing into, and displacing a part of the air?

c. Examine the litmus solutions, and compare their color(?). What property has the solution of carbon dioxide in water? How is the substance formed(equation)?

d. Make a list of the properties observed: Color(?), odor(?), density(?), diffusibility(?), solubility(?), supports combustion or not(?), what chemical action on water(?). Note which of these properties are physical and which chemical(?).

e. Why will any other acid act on marble as does hydrochloric acid [202, par. 1].

If you obtained a carbonate as an unknown substance, how should you proceed to identify it?

EXERCISE 31.

CARBON MONOXIDE.

Object: *To prepare carbon monoxide and to observe its properties.*

Apparatus: Flask(200 c.c.) with 2-hole stopper. Funnel, pinchclamp, straight tube and rubber connection. L-tube and delivery tube.

Trough. 3w.-m. bottles(250 c.c.), 3 glass plates.

Materials: Sulphuric acid(conc.). Formic acid. Limewater. Taper.

Caution: The gas is poisonous. Do not allow it unnecessarily to escape into the room.

a. Fit a 200 c.c. flask with a 2-hole stopper, L-tube and rubber delivery tube, and the funnel and straight tube with pinchclamp shown in Fig. 13(Ex. 25). Place the flask on the ring of the iron stand(with wire gauze), at a suitable height for heating by a small flame. Hold the funnel erect in the clamp. Extend the delivery tube, if necessary, by adding a bent glass tube so that the gas may be collected over water. Remove the stopper, pour into the flask about 1 cm. depth of concentrated sulphuric acid, and replace the stopper. Pour some formic acid into the funnel, and never allow this entirely to run out during the experiment.

Warm the sulphuric acid gently (small flame), and then admit the formic acid a drop or two at a time. Collect three bottles full of the gas, and immediately removed the delivery tube from the water, to avoid ascent of the water into the sulphuric acid (danger). What will the first bottle largely contain? Do not use this one. Do not wash out the apparatus until the acid has cooled (danger).

b. Light a taper, raise one bottle out of the water and plunge the taper up into it. Does the gas burn? Does the taper burn in the gas?

c. Slide the glass plate, for a moment, partly off a second bottle of the gas and pour in a little limewater. Shake(?). Now light a taper, set fire to the gas in this bottle, replace the cover instantly, and shake again(?). What product is formed when carbon monoxide burns? Make the equation(?).

d. Summarize the properties of carbon monoxide: color(?), solubility in water(?), burns or not(?), supports combustion or not(?). What is the density compared with that of air [175, 176]?

How could you distinguish burning hydrogen from burning carbon monoxide?

EXERCISE 32.

HYDROCARBONS.

Object: *To make two typical hydrocarbons, to ascertain their properties, and to compare the luminosities of their flames.*

Apparatus: Test-tube. 1-hole stopper and L-tube. 250 c.c. bottle 12" rubber tube. Evaporating dish. Beaker.

Materials: Soda lime. Sodium acetate. Limewater. Litmus papers. Hydrochloric acid (conc.). Calcium carbide.

a. Methane. Mix about 2 c.c. each of powdered soda lime and sodium acetate and place the mixture in a test-tube. Clamp this in a horizontal position and insert in it a 1-hole stopper with L-tube pointed upwards. Tap the test-tube to cause the mixture to settle and heat it gently (small flame). Slip a test-tube over the vertical part of the L-tube. When the gas in this test-tube, on being lighted (after removal to a distance), burns quietly, set fire to the jet issuing from the apparatus.

b. Describe the structure of the flame (?). How luminous is the flame? Hold a bottle so that the flame burns inside it. If a dew appears in the bottle, what is it? Inference (?). Withdraw and close the bottle, and shake the contents with some limewater (?). Inference (?).

Extinguish the flame, and test the gas for acid or basic properties by holding in it moistened strips of red and blue litmus paper (?).

c. Summarize the observed physical and chemical properties of the gas (?). Compare the density with that of air [176] (?).

d. Place some limewater in a test-tube. When the apparatus used in **b** has become cold, attach a rubber tube, add 5 c.c. of concentrated hydrochloric acid, replace the stopper, and let the issuing gas bubble through the limewater (?). Inference (?). What, beside methane, is the other product of

the reaction? Write the equations for the original action(?) and for the action of the acid on the product(?).

e. **Acetylene.** Fill a test-tube with water. Invert it in an evaporating dish partly filled with water. Place a small piece of calcium carbide in the water, and collect the gas in the test-tube.

Set fire to the gas in the test-tube. Compare the luminosity of the flame with that of methane(?). Close the tube, add a little limewater and shake(?). Inference(?).

f. Summarize the observed physical and chemical properties of the gas(?). Compare its density with that of air [176](?).

g. Pour the liquid in the evaporating dish into a beaker(?). Test it with litmus papers(?). What is the suspended solid? Write the equation for the interaction of the carbide and water(?).

EXERCISE 33.

FLAME.

Object: *To learn the source of the luminosity, and the structure of a flame. To find out the use of the holes at the base of a Bunsen burner. To observe the nature of the flame of a taper.*

Apparatus: Evaporating dish.

Materials: Splints. Calcium carbonate(pulv.).

a. Unscrew the tube of the Bunsen burner, turn on the gas, and light it(?). Turn off the gas and replace the tube. Make a drawing of the vertical section of the burner through the feed pipe(?).

Light the gas again and hold one side of the evaporating dish, first in the nonluminous flame and then in the luminous one(?). Which substance causes the light(?) and how? Why does this substance remain for a time suspended in the flame, instead of burning at once?

b. Hold a splint across the luminous flame close to the

bottom, and examine the wood(?). Inference(?). Repeat with the non-luminous flame(?). Why is the interior of the flame cold?

c. Is any gas entering or escaping from the holes at the base? To answer this, light a splint, blow the flame out, and hold the smoking end close to the holes(?). Confirm by scattering a very small amount of pulverized calcium carbonate on a piece of paper, and shaking it off so that it falls past one hole. Observe the flame(?).

Hold the burner up and blow a sudden puff of air towards one of the holes(?). What is the effect of increasing the supply of air [278]?

d. Light a taper and a splint. Blow out the flame of the taper and instantly apply the flame of the splint to the ascending "smoke"(?). Repeat, noting whether the taper can be relighted at a distance from the wick(?). Explain. Is the flame of a taper a gas flame or not?

EXERCISE 34.

STARCH AND SUGARS.

Object: *To study the properties of starch and its hydrolysis into glucose; and to learn the properties of the sugars, glucose and sucrose (ordinary sugar).*

Apparatus: Test-tubes. Flask. Funnel. Beaker. Glass rod. Graduated cylinder.

Materials: Fehling's solution No. 1 and No. 2.* Starch. Hydrochloric acid (conc.). Potassium iodide (sol.). Iodine. Sodium carbonate (sol.). Litmus papers. Sugar. Molasses.

a. **Starch and Glucose.** Shake about 0.5 c.c. of starch with 20 c.c. of water and then boil(?). Add a few drops of the

* No. 1: 69.3 g. of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ with water to make 1 liter. No. 2: 350 g. Rochelle salt and 100 g. sodium hydroxide with water to make 1 liter. Mix equal volumes (measured) just before use.

liquid to 5 c.c. of Fehling's solution * and warm the latter(?). Does starch change Fehling's solution?

Place three-fourths of the remaining starch suspension in a flask, set it on the wire gauze, add 5 or 6 drops of concentrated hydrochloric acid, and boil gently for ten minutes. Place the funnel in the mouth of the flask, to diminish the loss by evaporation, and add a little water when necessary so as to keep the volume constant.

b. While this is going on, cool the rest of the starch suspension in running water and pour it into a beaker full of cold water. Add one drop from 5 c.c. of a solution of potassium iodide in which a single crystal of iodine has been dissolved(?).

c. Cool the contents of the flask(from a) and add sodium carbonate solution drop by drop, shaking between drops, and touching the edge of a piece of litmus paper with a glass rod dipped in the liquid, until the liquid gives faint alkaline reaction.

Now add a few drops of this liquid to 5 c.c.(measured) of Fehling's solution and warm the latter [284, 647](?). Add more of the liquid, boil again, and continue until the blue color is gone. It takes 0.005 g. of glucose to reduce and decolorize 1 c.c. of Fehling's solution. What amount of glucose was contained in the part of the liquid you added?

d. **Sucrose.** Dissolve about 0.5 g. of sucrose(ordinary sugar) in 20 c.c. of water and repeat a and c with this liquid. Does sucrose reduce Fehling's solution? Do acids hydrolyze it to give glucose? Write the equation. The hydrogen-ion of the acid acts as a contact agent. Why is it not included in the equation?

e. Dissolve about 0.5 c.c. of molasses in water and test for glucose as in a, par. 1(?).

* See footnote on page 55.

f. Tabulate the physical and chemical properties of starch, glucose, and sucrose separately as follows: color(?), solubility(?), action on Fehling's solution(?), action of dilute acids(?).

EXERCISE 35.

FERMENTATION.

Object: *To ferment some molasses and to study some of the properties of alcohol.*

Apparatus: Large bottle(2 l.) or flask, 1-hole stopper to fit, with L-tube, delivery tube and glass tube. Distillation apparatus(Fig. 11, Ex. 11). W.-m. bottle. Mortar.

Materials: Molasses. Yeast. Limewater. Litmus papers. Asbestos wool. Sodium hydroxide(sol.). Iodine.

a. Mix 250 c.c. of molasses with 1500 c.c. of water in a bottle or large flask. Break up a cake of yeast, shake it with water until it is completely suspended, and add it to the molasses. Plug the mouth of the bottle loosely with cotton, and set the whole in a warm place for several days. This quantity will serve for 10-15 students. For individual experiments, take one-tenth of the above amounts.

b. Fit to the bottle a 1-hole stopper, L-tube, and rubber tube terminating in a straight glass tube. Take 15 c.c. of limewater in a test-tube, insert the glass tube to the bottom of this, and shake the fermented liquid round in the bottle(?). Note the gas evolved and its reaction with limewater(?). In what form of solution was the gas before the shaking [130]?

c. Filter off 100 c.c. of the fermented liquid, place it in a flask and distil as in Fig. 11, Ex. 11 a. After 30 c.c. has come over, empty and wash the flask, and re-distil the distillate until 6-8 c.c. has come over.

Note the odor of the distillate(?). Test it with litmus papers(?). Saturate a tuft of asbestos wool with the liquid, set the tuft on the base of the stand, and apply a light(?).

Hold a cold, w.-m. bottle over the flame, to recognize one product(?). Close the bottle and test the gas in the bottle by shaking with limewater(?). Make the equation for the combustion(?). If you burned alcohol vapor and pure oxygen, what relative volumes should you use, and what relative volumes of the products(measured as gases) would be formed [180]?

d. To a few drops of the alcohol add 1 c.c. of sodium hydroxide solution. Pulverize one or two(not more) crystals of iodine, add a part of the powder to the mixture and warm(?). The amount added must not be sufficient to give a permanent brown tint to the liquid. The precipitate is iodoform CHI_3 (related to chloroform CHCl_3).

e. Summarize the observed physical and chemical properties of alcohol(?).

EXERCISE 36.

ACETIC ACID.

Object: *To observe the properties of acetic acid, the difference between an active and an inactive acid, and the liberation of acetic acid from an acetate.*

Apparatus: Test-tubes.

Materials: Acetic acid(dil.6N). Sulphuric acid(dil.). Zinc(dust). Sodium acetate. Litmus papers.

a. **Acetic acid.** Take 5 c.c. of acetic acid and note its odor(?). Test it with litmus papers(?).

To show that acetic acid is a weak or inactive acid [213], take 15 c.c. of water in each of two test-tubes. To one add the 5 c.c. of acetic acid and to the other add an equal volume(5 c.c.) of dilute sulphuric acid. Add a little zinc dust to the contents of each tube, and compare(?). Which reaction goes faster?

If acetic acid had been added to the starch(Ex. 34 a) or sucrose(Ex. 34 d), instead of hydrochloric acid(a very ac-

tive acid), what change in the procedure would have been necessary to get the same result?

b. To about 0.5 c.c. of sodium acetate add 2 c.c. of water and 1 c.c. of concentrated sulphuric acid. Warm and note the odor(?). Test the vapor with litmus papers(?). Make the equation, the sodium forming NaHSO_4 (?).

c. Summarize the properties of acetic acid(?).

EXERCISE 37.

PERCENTAGE OF OXYGEN IN AIR.

Object: *To find the volume of oxygen contained in a definite volume of air by absorbing the oxygen out of this air and measuring the shrinkage.*

Apparatus: See Fig. 14. One hole in the cork (better than rubber stopper, because latter tends to slip out when wet with the alkali) is plugged with a bit of glass rod with fire-polished ends. The lower end of the short glass tube is previously heated until the opening shrinks to half its original diameter. Graduated cylinder.

Materials: Potassium pyrogallate (alkaline sol.*). Rubber bands. Taper.

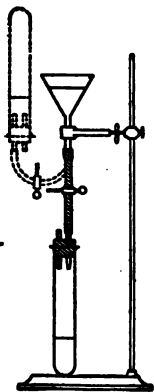


FIG. 14

a. Before attaching the funnel, test the apparatus (Fig. 14) for air-tightness.

Remove the test-tube, temporarily, and take the glass rod out of the cork. Pour about 40 c.c. of the potassium pyrogallate solution into the funnel. The following operations must be done quickly as the solution gradually absorbs oxygen from the air of the room and becomes useless. Do not get the solution on the hands, as it stains the skin.

Open the pinchclamp and permit the liquid to fill the rubber tube and the glass tube completely down to the opening of the nozzle. Replace the test-tube, fitting the cork tightly

* For 20 students, dissolve 200 g. of potassium hydroxide and 20 g.

into its mouth. Finally, close the hole in the cork with the glass rod, thus enclosing a volume of air equal to the capacity of the test-tube.

Now, open the pinchclamp. A few drops of the solution will run into the test-tube and, as the oxygen is absorbed, more will follow. Close the clamp and turn the test-tube upside down once or twice to bring the liquid near every part of the oxygen. In doing this, hold the test-tube with a folded strip of paper (Fig. 1, Ex. 1 a), and not with the bare hands, since this would warm the gas and render the result inaccurate. Then hold the clamp open, invert the test-tube and hold it so that the levels of the liquid in it and in the funnel are the same, and close the clamp.

Restore the test-tube to its original position and place on it two rubber bands to mark the levels of the lower end of the stopper and the surface of the liquid.

Light a taper, disconnect the test-tube, and plunge the flame into it(?). Without disturbing the bands, wash the test-tube out. By means of a graduated cylinder, measure and record the volumes of water required to fill the test-tube up to the lower(?) and to the upper(?) bands. The former is the volume of the oxygen, the latter that of the air.

Calculate the percentage of oxygen by volume in air as follows:

$$\frac{\text{volume of oxygen} \times 100}{\text{volume of air}} = x = \% \text{ oxygen.}$$

What gases were left, after the oxygen had been absorbed?
What properties of nitrogen did you observe?

pyrogallie acid in 1 liter water immediately before use and keep tightly stoppered.

EXERCISE 38.

CARBON DIOXIDE IN AIR AND IN THE BREATH.

Object: *To show the presence of carbon dioxide in atmospheric air and in expired air.*

Apparatus: Beaker. Glass tube. Test-tube. 2 w.-m. bottles and glass plates. Trough.

Materials: Barium hydroxide(sol.). Taper. Limewater.

a. Place about 3 c.c. of clear barium hydroxide solution in the bottom of a clean beaker and leave it exposed to the air for half an hour or more(?). Barium hydroxide $\text{Ba}(\text{OH})_2$ behaves towards carbon dioxide like limewater, but, being more soluble than calcium hydroxide, its solution is more concentrated and a more copious precipitate can be obtained. Explain the result(?), and write the equation for the action(?).

b. Blow air from the lungs through a straight tube into 5 c.c. of limewater(?).

c. Fill two bottles with water and invert them in the trough. By means of a tube, fill one with air from the lungs immediately after drawing a breath. Fill the other with air after the lungs have been almost emptied. Slip a glass plate under each bottle and set both upright on the table. Light a taper and plunge it into one bottle(?) and then into the other(?). Explain the result(?).

EXERCISE 39.

AMMONIA.

Object: *To study two ways of obtaining ammonia and to observe its physical and chemical properties.*

Apparatus: Test-tubes. 1-hole stopper, L-tube, and perforated cardboard. 2 w.-m. bottles, and glass plates. Glass rod. Trough. Beaker. Cork.

Materials: Gelatine(fine flakes). Litmus papers. Soda lime. Am-

monium chloride. Slaked lime. Ammonium sulphate. Hydrochloric acid (conc.).

a. From Organic Matter. Take 0.5 c.c. of gelatine in a dry test-tube. Place moistened litmus papers (blue and red) in the mouth of the tube and heat gently. Note the odor(?) and effect on litmus(?). Insert a glass rod wet with concentrated hydrochloric acid(?).

Repeat, first mixing on paper the gelatine with an equal amount of soda lime(?). Almost all organic compounds containing nitrogen give ammonia when heated with soda lime, and this is therefore a test for such compounds.

b. From an Ammonium Salt. Place a little ammonium chloride in the palm of one hand, a little slaked lime in the other, and rub the two together. Note the odor(?) and effect on moistened litmus paper held over the mixture(?).

Repeat, using ammonium sulphate and slaked lime(?).

c. Take one-fourth of a test-tube full of ammonium chloride and mix it on paper with an equal volume of slaked lime. Place it in a test-tube provided with a one-hole stopper and L-tube long enough to reach the bottom of the inverted bottle (Fig. 15). Clamp the tube so that the mouth is inclined very slightly downwards (to prevent condensed moisture running back). Arrange the perforated card on a ring, and first slip an inverted test-tube over the L-tube. Provide a test-tube containing a few drops of concentrated hydrochloric acid. Also, place water in the trough.

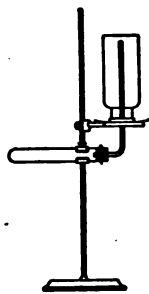


Fig. 15

Warm the mixture very gently with a small flame. Dip the end of a thin glass rod in the hydrochloric acid, and bring it close to the perforation in the card to ascertain when the test-tube is filled with the gas. Dense smoke will notify you of this condition. Remove the test-tube, closing it firmly

with the thumb, and with the other hand set a bottle(to be used in e) over the L-tube.

d. Place the mouth of the test-tube under water and remove the thumb(?). Is the gas soluble in water? How does its behavior compare with that of chlorine(Ex. 21 a) in this respect?

e. Warm a second bottle by moving it rapidly through the flame. Do not let it rest in the flame or it will crack. Place in the bottle 3 drops of concentrated hydrochloric acid, cover it with a glass plate, and turn it so as to spread the acid as completely over the inner surface as possible.

Now, ascertain by use of the rod dipped in hydrochloric acid whether the first bottle (c) is full of ammonia. When it is, raise it off the tube, slip a glass plate under the mouth, and set the bottle mouth downwards on the table ready to be used in g.

f. Swing the L-tube downwards so that its free end is just above(but not touching!) the surface of the 5 c.c. of water in the bottom of a beaker. At the end of 5 minutes pour the solution into a test-tube, cork it up, and reserve for use in Ex. 40. During the five minutes proceed with g, but, swing the water round in the beaker occasionally to submerge the surface layer. Concentrated ammonia [323] has a density of 0.88, i.e. 1 c.c. weighs 0.88 g. Will the solution float or sink? What difference would it make if the beaker were left at rest?

g. Bring the two bottles from e mouth to mouth and remove both plates. Invert the pair of bottles once or twice to mix the gases(?). What is the deposit? Describe its physical properties(?). Write the equation for the action(?).

h. Summarize the observed properties of ammonia(?).

EXERCISE 40.

HYDROXIDE AND SALTS OF AMMONIUM.

Object: *To observe the properties of ammonium hydroxide and of salts of ammonium, and to learn a test for the latter.*

Apparatus: Glass rod. Evaporating dish. Beaker. Test-tubes.

Materials: Litmus papers. Hydrochloric acid (dil.). Ammonium chloride. Sodium hydroxide (sol.). Ammonium sulphate.

a. **Ammonium Hydroxide.** Dip a rod in the solution made in Ex. 39 f and touch litmus papers with it(?). To what class of substances does the material in solution belong? Write the equation for the formation of this material(?).

b. Pour a part of the solution into an evaporating dish and boil it. From time to time observe its odor and reaction towards litmus papers(?), proceeding meanwhile with c. What must have happened to the compound in the solution? Is it a stable or an unstable compound [30]? Write the equation(?). Is this action a reversible one or not? Is it complete?

c. Place the rest of the solution in a beaker, add dilute hydrochloric acid a little at a time, stirring with a rod and testing the liquid on litmus papers until the liquid is neutral. Evaporate the liquid in a dish almost (but not quite) to dryness (lower the flame towards the end), allow it to cool and describe the residue(?). Write the equation(?).

d. Summarize the properties of ammonium hydroxide(?).

e. **Ammonium Salts.** Place not over 0.2 c.c. of ammonium chloride in the bottom of a dry hard glass test-tube. Clamp the latter in a horizontal position, and place in the mouth of the tube moistened litmus papers (red and blue). Heat the salt and watch the test papers for any changes and describe them(?). The second change takes much time.

Is there evidence that the salt decomposed? What were presumably the products? Make the equation(?). Would these products recombine (partially at least) in the cool part

of the tube(Ex. 39 g)? Is there any sublimate visible(?) and if so, what is it?

What would be the final effect of continuing the heating of the salt?

f. Test for Salts of Ammonium. Take 2 c.c. of sodium hydroxide solution and add a few particles of any salt of ammonium(*e.g.* am. sulphate). Warm, and observe the odor(?).

Write the equation as a double decomposition(?). Which of the products decomposes to give the ammonia?

EXERCISE 41.

FORMS OF SULPHUR.

Object: *To study a substance which shows two solid forms, and two liquid forms.*

Apparatus: Test-tubes. Mortar. Watch glass. Lens (1 for 10 pupils). Trough. Funnel.

Materials: Sulphur(roll). Carbon disulphide. Filter paper.

a. Rhombic Sulphur. Place about 0.5 c.c. of powdered roll sulphur in a dry test-tube, add 2 c.c. of carbon disulphide and shake(?). Pour the solution into a watch glass and set it to evaporate away from all flames.

Examine the crystals with the eye and with a lens and make a drawing of two of them(?). Are they brittle or soft? Color? Are they transparent? After 24 hours(or more), are they still transparent?

b. Monoclinic Sulphur. Fold a filter-paper as if for filtration, and put water in the trough. Half fill a dry test-tube with roll sulphur. Hold the test-tube with the clamp from the iron stand and melt the sulphur. Heat gently, turning the tube in the flame. Dark brown patches show overheating at these points—melted sulphur is pale straw-color.

Hold the filter paper by the edge at the three-fold side and pour the melted sulphur into it. Watch the crystals

grow. When crystals have formed at the surface and reached the center, pour the remaining liquid sulphur promptly into the trough of water, and open up the paper immediately.

Examine the crystals with the eye and with the lens and make a drawing of two of them(?). Are they brittle or soft? Color? Are they transparent? After 24 hours(or more), are they still transparent? If not, into what form of sulphur have they turned?

Examine the part that was poured into water. Is it brittle or soft? Dry a small piece of it and shake it with 1 c.c. of carbon disulphide(?).

c. **Amorphous Sulphur.** Half fill the same test-tube with roll sulphur and melt. Continue heating until the sulphur boils and note the changes in color and fluidity which occur(?). If the vapor catches fire, allow it to burn.

Pour the boiling sulphur into the trough of water, moving the tube about.

Examine the product. Is it crystalline? Is it brittle or soft? Is it transparent? After 24 hours(or more) is it still transparent? After 24 hours(or more), dry a part of it and shake with a little carbon disulphide(?). To find out whether any has dissolved, pour part of the liquid on to a filter, catch a few drops of the filtrate on a watch glass, and allow them to evaporate(?).

What is the insoluble material? Color? Examine with a lens(?). Is it crystalline?

EXERCISE 42.

SULPHIDES OF METALS.

Object: *To observe the activity of sulphur towards metals.*

Apparatus: Test-tubes. Mortar.

Materials: Sulphur(roll). Copper foil(strips). Iron(powder). Mercury(dropper in bottle).

a. Place about 2 c.c. of roll sulphur in a dry test-tube

and fasten the tube upright in the clamp on the stand. Boil the sulphur and then drop into the tube a strip of copper foil(?). Does the temperature of the copper change noticeably? Was it heated thus by the flame, the sulphur vapor, or what?

Remove the strip of copper and describe the properties of the product(?). Name it(?) and write the equation(?).

b. Mix thoroughly 1 c.c. of iron powder with 1.5 c.c. of powdered roll sulphur. Place the mixture in a test-tube and clamp the latter on the stand. Heat the lower part of the material and, when the reaction begins, withdraw the flame.

Was the temperature at any time higher than that which the flame could have produced? What was the source of the heat?

Describe the product when cold(?). Name it(?), and write the equation(?). Keep it for use in Ex. 43 e.

c. With a dropper place a single globule of mercury in the mortar. Add an equal volume of sulphur and rub the two for several minutes, or until no mercury is visible(?). Under the same conditions (pressure, temperature, contact), does mercury combine more or less readily with sulphur than with oxygen? Give reasons for your answer(?).

Does sulphur appear to have about the same activity as oxygen, or much more or much less?

EXERCISE 43.

HYDROGEN SULPHIDE.

Object: *To prepare hydrogen sulphide. To study its physical and chemical properties, and to consider uses for them.*

Apparatus: Test-tube with 1-hole stopper, L-tube, and rubber tubing. W.-m. bottle and glass plate.

Materials: Ferrous sulphide (small lumps). Hydrochloric acid (dil.). Litmus papers. Cupric sulphate (sol.). Arsenious chloride* (sol.). Lead nitrate (sol.). Antimony trichloride (sol.).

* Dissolve arsenic trioxide in dilute hydrochloric acid.

a. Take a test-tube fitted with a 1-hole stopper, L-tube, and rubber tube ending in a straight glass tube. Hold the test-tube almost horizontal and slip into it carefully about 3 c.c. of ferrous sulphide. Add dilute hydrochloric acid and replace the stopper. Note the color(?) and odor(?) of the gas. Avoid breathing it, however, as far as possible.

b. Fill a bottle with the gas by upward displacement of the air and cover the bottle with a glass plate.

Set fire to the gas at the end of the delivery tube. Odor? What products are formed? Place the delivery tube in a test-tube half filled with water, so that it reaches the bottom and let the gas bubble through the water for several minutes. Use the solution in c.

Now set fire to the gas in the bottle(?). What products are formed when the gas burns in a bottle? Why is the result different from that when a jet of the gas burned in the air?

When the solution has been made, pour water into the generating tube and wash it out. Put solid matter in the jar—not in the sink.

c. Smell the solution made in b(?) and test it with litmus paper(?). To what class of substances does hydrogen sulphide belong? What other name is given to the solution?

Take 4 test-tubes and in each place about 2 c.c. of a solution of a different one of the following salts: (1) Cupric sulphate, (2) Arsenious chloride AsCl_3 , (3) Lead nitrate $\text{Pb}(\text{NO}_3)_2$, (4) Antimony trichloride SbCl_3 . Add a part of the solution of the gas to each(?).

What variety of chemical change will an acid and a salt undergo in solution [202, 1]? Make equations for the four actions accordingly(?).

d. Summarize the observed properties of hydrogen sulphide(?). How could you identify hydrogen sulphide?

How could you use it in identifying the metallic radicals in different salts?

- e. To the residue from Ex. 42 b, add dilute sulphuric acid. Identify the gas liberated, using at least three different properties, and record the results(?).

EXERCISE 44.

SULPHUR DIOXIDE.

Object: To prepare sulphur dioxide and to study its properties.

Apparatus: Watch glass. Platinum wire. Wide test-tube, 2-hole stopper, funnel, pinchclamp, rubber and glass tubing. Test-tubes. W.-m. bottle, glass plate and cork. Trough.

Materials: Sulphur. Sodium bisulphite. Hydrochloric acid (dil.). Pinks or grass or leaves. Apple.

a. Place on a watch glass a particle of sulphur and touch it with a warm platinum wire. Bring the wire with the adhering sulphur again into the flame. Withdraw and note the color(?) of the flame of burning sulphur and the odor(?) of the gas produced. Name the gas that has this odor(?), and write the equation(?).

b. Fit up an apparatus as in Fig. 13 (Ex. 25, II a). Place in the test-tube about 10 c.c. of sodium bisulphite,* and attach a straight glass tube reaching down to the bottom of an upright, dry test-tube. Put dilute hydrochloric acid in the funnel, and admit it drop by drop to the apparatus. If there is little evidence of action (bubbling), the apparatus may be warmed very slightly to start the reaction.

Put water in the trough. Also have ready a bottle containing a moist pink (or some grass or leaves) and half a slice of an apple. Leave the other half-slice exposed to the air for comparison later. Likewise boil half a test-tube full of water for 2 minutes and set aside to cool.

* Heating copper with sulphuric acid involves unnecessary risks.

c. Now transfer the delivery tube to the bottle, and close the mouth of the test-tube firmly with the thumb. Pour quickly a little water into the test-tube and close again firmly. Still keeping the thumb in position, place the mouth of the test-tube under the water in the trough. Notice the level of the water in the test-tube and then remove the thumb(?). Inference(?).

d. Cool the boiled water in the test-tube, and again transfer the delivery tube, inserting it to the bottom of boiled water, and let the gas run in for 5 minutes. Cork this tube the instant the delivery tube is taken out, so as to enclose the gas, and not admit air. Keep this solution for Ex. 45.

Cover the bottle or, better still, cork it. After 5 minutes, pass more of the gas into the bottle, and finally cork it again. Observe the color of the contents from time to time(?).

Compare the pieces of apple after both have been exposed to the air for a day or more(?).

e. Summarize the properties of sulphur dioxide: color(?), odor(?), density compared with air [175, 176](?), solubility(?), action on vegetable coloring matters(?).

✓ EXERCISE 45.

SULPHUROUS ACID.

Object: *To learn the properties of sulphurous acid.*

Apparatus: Evaporating dish. Test-tubes. Mortar.

Materials: Sulphurous acid (from Ex. 44 d). Litmus papers. Barium chloride(sol.). Iodine.

a. Use the solution made in Ex. 44 d. Test with litmus papers(?). What sort of substance has been formed? Is sulphur dioxide an acidic or a basic oxide [349]?

b. Pour a part of the solution into an evaporating dish, set it on the wire gauze, and boil it [HOOD], noting the odor from time to time(?). Is sulphurous acid stable?

note c. To half of the rest of the solution add one or two drops of barium chloride solution(?). Disregard any very slight cloudiness.

To the other half add a couple of crystals of iodine(pulverized in the mortar), shake until the iodine has dissolved, and then add barium chloride BaCl_2 solution(?). The precipitate is barium sulphate BaSO_4 , and its formation shows that the radical SO_3 has become SO_4 .

Read [355]. Which substance was here oxidized?

What substance was reduced? Write equations for the action of iodine(?), and the subsequent action of barium chloride(?).

d. Summarize the properties of sulphurous acid: color(?), odor(?), stability(?), oxidizing or reducing agent(?).

If the water for the solution had not been boiled, what would have happened [42, 355, 127, par. 3] ?

How should you prepare a solution of sodium sulphite from sulphurous acid?

✓ EXERCISE 46.

SULPHURIC ACID.

Object: *To learn the properties of pure and of diluted sulphuric acid, and to try a test for the sulphate radical.*

Apparatus: Test-tubes. Glass rod. Evaporating dish.

Materials: Sulphuric acid(conc.). Litmus papers. Barium chloride(sol.). Hydrochloric acid(conc.). Sodium sulphate. Copper (shavings).

a. Take 5 c.c. of water and pour into it about 1 c.c. of concentrated sulphuric acid [CAUTION. Never pour the water into the acid]. Touch the back of the hand with the bottom of the test-tube(?).

b. Apply the solution with a glass rod to litmus papers(?).

note c. Boil nearly all the rest of the solution gently in an evaporating dish with a small flame and note whether there is

any odor. After five minutes, test again with litmus papers(?). Is the acid like sulphurous acid, or is it more stable(?). Let the dish cool before washing it out.

d. Test for the sulphate radical SO_4 . To the remainder of the solution add barium chloride solution(?). Add now 1 c.c. of concentrated hydrochloric or nitric acid(?). Other common salts of barium, if precipitated, would be decomposed and dissolved by these acids. Write the equation(?).

Repeat the test, using a few particles of sodium sulphate or ammonium sulphate dissolved in water(?). Write the equation(?).

e. Twist a few copper shavings into a ball and place them in a test-tube. Add 1 c.c. of concentrated sulphuric acid. Heat gently with a small flame. Note how long it takes to make the acid hot enough to interact at all [CAUTION! Hot sulphuric acid, if spilled, may produce severe burns]. Note the odor(?). Test to see whether any hydrogen is produced(?). Has the sulphuric acid been reduced? What sort of substance is hot concentrated sulphuric acid?

f. Summarize the properties of the acid: State(?), color(?), odor(?), stability(?), litmus(?), action on water(?), on barium chloride(?), on elements like copper(?).

EXERCISE 47.

RECOGNITION OF SUBSTANCES I.

(Mainly certain negative radicals.)

Object: To learn how substances may be recognized by their properties, and to review the methods of preparation and properties studied in previous exercises.

Apparatus: Test-tubes. Glass rod. Watch glass.

Materials: Unknowns(Appendix VI). Litmus papers. Filter paper. Sodium hydroxide(sol.). Limewater. Sulphuric acid(conc.). Fehling's solution(I and II). Iodine(sol. in KI). Barium chloride(sol.). Hydrochloric acid(conc.).

a. Obtain [Instructor] a single unknown solid substance. This will contain one of the following radicals:

ammonium(NH_4)	sulphite(SO_3)
chloride(Cl)	sulphate(SO_4)
carbonate(CO_3)	formate(CO_2H)
acetate(CO_2CH_3)	base(OH)
sulphide(S)	

It may, however, be starch, sucrose, glucose, or sulphur.

In the case of a salt(except one of ammonium) we shall limit ourselves to identifying the negative radical only. The other positive radicals will, in any case, be limited to(Na), (K), and(Ca).

Record at the time the result of each observation. Record negative results also.

b. **External Examination** [485]. Begin by recording the state(?), crystalline form(?), color(?), and odor(?).

c. **Solubility and Reaction of Solution.** Use a few particles to find out whether it is soluble in water(?). If in doubt, proceed as in Ex. 10 f. Apply a drop of the solution to litmus papers(?).

d. **Effect of Heating** [486]. Heat 0.5 c.c. in a dry test-tube. Watch the substance. Does it melt(?), char(?), or otherwise change(?). Note also gases or vapors.

If WATER VAPOR is given off and condenses(?), the substance may be a hydrate, starch or one of the sugars, or calcium hydroxide. Incline the tube downwards, drive all the water off, dry out the tube with filter-paper, and continue heating.

Does it now char(?), change(?), or give gases or vapors?

If a **SUBLIMATE**(solid deposit in the tube) appears(?), it is a salt of ammonium. Note the odor(?) and apply the test (Ex. 40 f).

A COLORLESS GAS with an ODOR(?) may be ammonia, sulphur dioxide, or hydrogen sulphide, or gases from the charring of carbohydrates(see e).

A COLORLESS GAS with NO ODOR. Examine for carbon dioxide by inserting a rod dipped in limewater(?). If you get a positive result, what was the unknown substance [249, par. 3] ?

e. **Effect of Sulphuric Acid [487].** To 0.5 c.c. of the substance add two or three drops(not more) of concentrated sulphuric acid(?). If necessary, warm gently(?). Watch the substance, and look out for gases.

If the substance turns BLACK, it is a carbohydrate. Identify it by fact that starch(insoluble) gives the iodine test (Ex. 34 b), glucose reduces Fehling's solution(Ex. 34 c), and sucrose does not(Ex. 34 d).

A COLORLESS GAS(bubbling), which FUMES in the breath, is hydrogen chloride. If it does fume, what was the substance [139, 140] ?

A COLORLESS GAS which does NOT FUME, may have an ODOR(?). If the odor is that of hydrogen sulphide, what do you infer [339] ? If it is that of sulphur dioxide, inference [351, par. 4](?). If it is that of acetic acid, inference(Ex. 36 b)(?).

A COLORLESS GAS, which does NOT FUME and is ODORLESS, may be carbon dioxide. Test with rod dipped in limewater(?). Inference [249, par. 1](?). It may be carbon monoxide. Try whether it is combustible(?). Inference [253](?).

If there is NO BUBBLING, there is no gas. The substance may be a sulphate. Test(Ex. 46 d)(?). It may be a base (test?).

f. **Report.** State your conclusion, with the reasons therefor(?).

g. Obtain a second unknown and proceed as before(?).

EXERCISE 48.

HYDROGEN PEROXIDE.

Object: *To prepare a solution of hydrogen peroxide, to observe its properties, and to try a delicate test for its presence.*

Apparatus: Glass rod. Flask. Test-tubes. Funnel.

Materials: Sodium peroxide(pulv.). Litmus papers. Sulphuric acid (dil.). Manganese dioxide. Splints. Lead nitrate(sol.). Ammonium sulphide(sol.). Filter paper. Potassium dichromate (sol.). Ether.

a. To prepare a solution of hydrogen peroxide, take 100 c.c. of cold water in a flask, and about 1 c.c. of pulverized sodium peroxide on a piece of paper. Add the peroxide to the water, a very little at a time, shaking and cooling the mixture in running water during the process.

Test a drop of the solution on litmus papers(?). Now, while still shaking and cooling in the same way, add dilute sulphuric acid a few drops at a time, until the mixture is acid. Write the equation(?). What does the solution now contain?

b. Take 15-20 c.c. of the solution in a test-tube, and place about 1 c.c. of pulverized manganese dioxide on a piece of paper. Light a wooden splint, throw the manganese dioxide into the solution(?), and test the gas in the tube for oxygen(?). Write the equation(?). The manganese dioxide (catalyst) is unchanged. Is hydrogen peroxide stable or not?

c. Take 2 drops of lead nitrate solution $\text{Pb}(\text{NO}_3)_2$, dilute with 5 c.c. of water and add 2 drops of ammonium sulphide solution $(\text{NH}_4)_2\text{S}$ (?). Pour the mixture on to a filter. The precipitate is lead sulphide, formed by double decomposition. Write the equation(?). Wash the precipitate and whole filter paper with water and, when the water has run through, wash once more. Then pour upon the precipitate some of the hydrogen peroxide solution [373, par. 3](?). Write the equa-

tion(?)? To what class of substances does hydrogen peroxide here show itself to belong?

d. Test for Hydrogen Peroxide. Take about 1 c.c. of potassium dichromate solution and, to liberate dichromic acid, add an equal volume of dilute sulphuric acid. Take now a portion of the hydrogen peroxide solution, add 2-3 c.c. of ether, close with the thumb and shake. Then add to it one drop of the solution containing dichromic acid and shake again [373, par. 4](?).

e. Summarize the observed properties of hydrogen peroxide: color(?), solubility(?), stability(?), oxidizing or reducing action(?), test(?).

EXERCISE 49.

HYPOCHLOROUS ACID. BLEACHING.

Object: *To find out how the commonest "bleach" does its work, and what kind of substances it bleaches.*

Apparatus: 90 c.c. generating bottle, 2-hole stopper, thistle, L-, and delivery tubes. Funnel. Test-tubes.

Materials: Bleaching powder. Filter paper. Marble(chips). Litmus papers. Colored calico(small strips). Hydrochloric acid(dil.).

a. Take about 2 c.c. of bleaching powder with 20 c.c. of water in a test-tube, shake from time to time and finally filter to obtain a clear solution. Meanwhile arrange an apparatus to generate carbon dioxide as in Ex. 30 **a**. Pour most of the clear bleaching powder solution into another test-tube, and pass carbon dioxide through it for 5 minutes. The precipitate is calcium carbonate CaCO_3 . Filter again, to obtain a clear solution of hypochlorous acid [377] for use in **c**.

b. In the rest of the bleaching powder solution dip strips of litmus paper. Leave one in the solution(?). Does bleaching powder solution bleach? Hang the other in the air(?). What acid here liberates the hypochlorous acid? To what

class of chemical actions does that on the litmus belong?

c. In the hypochlorous acid solution from a place small pieces of (1) litmus paper, (2) paper with printing on it [277], (3) paper with writing in ink [616] and (4) in pencil [236], and (5) colored calico. Observe and record the effect on each(?). Which of these owe their color or blackness to free carbon, and which to colored organic compounds? Does hypochlorous acid oxidize free carbon? What does it oxidize?

EXERCISE 50.

NITRIC ACID.

Object: To prepare nitric acid, to study its properties, and to try a test for a nitrate.

Apparatus: Tubulated retort (glass stopper). Flask. Trough. Funnel. Porcelain crucible.

Materials: Sodium nitrate. Sulphuric acid (conc.). Charcoal (splinters). Woolen yarn (white). Copper (shavings). Splints. Zinc (gran.). Ferrous sulphate (sol.).

a. Put about 20 g. of sodium nitrate in a tubulated retort. Insert the neck of the retort into the mouth of a flask (Fig.

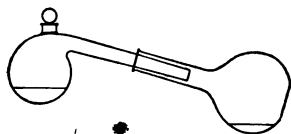


Fig. 16

16). Clamp the tubulus (mouth) of the retort so that the body rests on the wire gauze and the flask is partly immersed in a vessel of cold water. Through a funnel or thistle tube pour about

15 c.c. of concentrated sulphuric acid upon the sodium nitrate, place the stopper in the tubulus of the retort, and wait until the acid has moistened the entire mass.

Caution: Sulphuric acid and nitric acid both produce severe wounds on the flesh and destroy clothing. The nitric acid to be prepared is 100%, and the greatest care must be used in handling it.

Heat the retort very gently, and distil at as low a temperature as possible until no more nitric acid condenses in the neck of the retort(meantime, proceed with d). Allow the retort to cool before touching it. What is the brown gas seen in the retort(?), and whence does it come [388]? What is the residue in the retort?

b. Pour part of the distillate(nitric acid) into the porcelain crucible. Does its vapor produce a fog with moist air (breath)? Twist an iron wire round a splinter of charcoal, set fire to the latter, and dip the brightly glowing charcoal into the acid(?). This shows its oxidizing power. What compound of carbon is probably formed?

c. Divide the rest of the distillate between four dry test-tubes. In one place a piece of white woolen yarn(?), in the second a small piece of copper(?), in the third a piece of a splint(?), and in the fourth a small piece of zinc(?).

The action on copper is a test for nitric acid, by itself, or mixed with other substances.

What gas does zinc displace from other acids? What gas is produced, with zinc, here?

d. **Test for the (NO_2) radical**(follow the directions with care). Pour into a test-tube 3 c.c. of ferrous sulphate solution. Add to it 2 drops of dilute nitric acid and shake. Incline the tube very slightly, and pour concentrated sulphuric acid(not over 2 c.c.) in a steady stream down the wall of the test-tube so that(being a heavy liquid) it flows to the bottom and collects below the ferrous sulphate solution(?). Describe the coloration in the layer where the liquids meet(?).

Repeat the test, adding a small crystal of sodium nitrate, instead of the nitric acid, and shaking.

How should you recognize an unknown substance to be a nitrate?

e. Summarize the properties of nitric acid(?).

EXERCISE 51.

NITRIC OXIDE.

Object: *To observe the action of dilute nitric acid on copper. To prepare nitric oxide and study its properties. To observe part of the process used in the fixation of atmospheric nitrogen.*

Apparatus: Gas generating bottle (90 c.c.), 2-hole stopper, thistle, L-, and delivery tubes. Trough. W.-m. bottle. Graduated cylinder. Test-tubes. Deflagrating spoon. Glass plate. Wide test-tube, 2-hole stopper, dropper, L-, and delivery tubes.

Materials: Copper (shavings or clippings). Nitric acid (conc.). Asbestos paper. Taper. Phosphorus (red). Sodium peroxide. Litmus papers.

a. Fit up a generating bottle as in Fig. 10 (Ex. 10 a). Place in it copper shavings or clippings. Invert one bottle, one test-tube, and the graduated cylinder, all full of water, in the trough. Pour some water through the thistle tube, and an equal volume of concentrated nitric acid and wait for the action to start. Then fill the bottle and test-tube with the gas, collect about 30-50 c.c. (only) of the gas in the graduated cylinder, and leave them standing in the trough.

Note the color of the gas in the generator just after the beginning of the action (?) and again later (?).

Note also the color of the liquid in the generator. This is characteristic of cupric salts, and is the color of the cupric ion Cu^{++} .

b. What is the color of the gas? Use the test-tube full of the gas to test its solubility in water as in Ex. 44 c (?). What happens when the test-tube is opened to the air (this action is examined further in d)?

c. Line the deflagrating spoon with asbestos paper and place in it a little red phosphorus. Cover the bottle full of the gas with a glass plate, and set it upright on the table. Plunge a burning taper into the gas (?), withdraw it instantly, and cover the bottle. Set fire to the phosphorus, and

when it is burning vigorously, plunge it into the same bottle(?).

Heat the spoon and asbestos paper, to burn the adhering phosphorus, before putting the spoon away.

d. Fit a wide test-tube with a 2-hole stopper, L-tube and rubber delivery tube, and a dropper (Fig. 17). Place in the test-tube about 1 c.c. of sodium peroxide. Fill the dropper with water, and immerse the delivery tube in the trough. Pinch the dropper cautiously so as to allow one drop of water at a time to fall on the peroxide. When the air has been displaced from the test-tube, read the volume of the gas in the graduated cylinder(?), and allow the oxygen to ascend, a few bubbles at a time, into the latter(?). Note the change in color(?). Slightly shake the water in the cylinder to bring it in contact with the gas. Does the volume change(?) and, if so, in which direction? Remember that oxygen gas is being *added*.

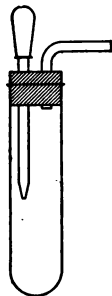


FIG. 17

Finally, close the cylinder with a glass plate, set it upright, and test the water in it with blue litmus paper(?).

e. Which component of the air caused the nitric oxide to become brown in b? What is the brown gas? What property of the brown gas have you learned in d? What reaction to litmus had the water in the cylinder(?) and what was the substance formed [394]? What great industrial process is based on this reaction [395]?

f. Summarize the properties of nitric oxide(?).

EXERCISE 52.

NITROUS OXIDE.

Object: To prepare nitrous oxide and to study its properties.

Apparatus: Test-tube, 1-hole stopper, L-, and delivery tubes. Test-tubes. W.-m. bottle. Trough. Deflagrating spoon.

Materials: Ammonium nitrate. Splints. Asbestos paper. Sulphur.

a. **Preparation of Nitrous Oxide.** In a large test-tube provided with a 1-hole stopper and rubber and glass delivery tube, place 10 g. of ammonium nitrate. Clamp the test-tube, mouth upwards, at an angle of 45° . Fill one bottle and two test-tubes with water and invert them in the trough (use warm water, if available).

Heat the nitrate cautiously but continuously with a small flame, and allow the gas to come very slowly. If, before the vessels are filled with gas, the nitrate threatens to give out [Explosion possible], first remove the delivery tube from the trough, then stop heating, and add more ammonium nitrate.

Watch the water in the delivery tube. Does anything else appear to be produced along with the nitrous oxide gas? What is it?

b. Use one test-tube of the gas, as in Ex. 44 c, to find out whether the gas is soluble in cold water(?). Does the gas differ from oxygen in this respect?

c. Into the second test-tube thrust a glowing splinter(?). Does the gas differ from oxygen in this respect?

d. Line the deflagrating spoon with asbestos paper and place on it a little sulphur. Set fire to the sulphur, without heating the spoon. Immediately, while it is still burning feebly, thrust it into the bottle of the gas(?), and then remove it and cover the bottle. Heat the spoon until the sulphur burns briskly and thrust it into the bottle once more(?). How does the gas differ in behavior from oxygen?

Note the odor in the bottle(?). What is formed when sulphur is burned in nitrous oxide?

e. Summarize the properties of nitrous oxide: color(?), solubility(?), density compared with air [175, 176](?), supports combustion, how well(?).

f. Name two respects in which nitrous oxide differs from oxygen(?). When nitrous oxide is added to nitric oxide, no brown gas(Ex. 51 d) is formed: this is another difference.

EXERCISE 53.

BROMINE.

Object: *To liberate bromine, to note its properties, and to try a test for bromides.*

Apparatus: Test-tubes! Mortar.

Materials: Potassium bromide. Sulphuric acid(conc.). Manganese dioxide(pulv.). Bromine. Carbon disulphide. Bromine-water. Chlorine-water.

a. Take 1 c.c. of water in a test-tube and add slowly 2 c.c. of concentrated sulphuric acid. Pulverize about 0.5 c.c. of potassium bromide, add 1 c.c. of manganese dioxide, mix, and add the mixture to the diluted acid. Warm very gently with a small flame(?). Note the color(?) and odor [CAUTION](?) of the vapor. What was the color of the potassium bromide? Does a compound show the colors of elements contained in it? Should you expect it to do so [10]?

If any of the bromine vapor is condensing on the sides of the tube, describe its properties(?). Fill the tube up with water. Write the equation [403](?).

b. To about 10 c.c. of water in a test-tube add two drops of bromine [CAUTION: Do not spill upon the hands (405)]. Is the bromine heavier or lighter than water? Shake. Is it soluble in water? Use this bromine-water in c.

c. To 10 c.c. of water add about 2 c.c. of carbon disulphide, close with the thumb, and shake(?). Are the liquids miscible(mutually soluble)?

To this mixture add a few drops of bromine-water from b and shake. Is the bromine, judging by the color, equally soluble in both water and disulphide? If more so in one, how much more(?) and in which?

d. **Test for a bromide.** Take a crystal of potassium bromide(color?) and dissolve it in 5 c.c. of water(color of solution?). Add 2 c.c. of carbon disulphide and shake(?). Why is the carbon disulphide not colored by the bromine?

Holding the tube steady, add now a drop or two of chlorine-water, and observe carefully whether any color appears and, if so, in which layer of liquid(water layer or carbon disulphide) it makes its appearance(?). Why? Shake vigorously and allow the mixture to settle(?). What substance has been liberated? Write the equation [403](?).

e. Summarize the properties of bromine: color(?), odor(?), density of vapor compared with air(?), density of liquid compared with water(?), relative solubility in water and carbon disulphide(?).

Has bromine or chlorine the greater affinity for potassium?

✓ EXERCISE 54.

IODINE.

Object: To prepare some iodine and use it to observe the properties of iodine. Also to try a test for iodides.

Apparatus: Evaporating dish. Mortar. 400 c.c. Beaker. Test-tubes.

Materials: Sulphuric acid. Potassium iodide. Manganese dioxide (pulv.). Alcohol. Carbon disulphide. Potassium iodide(sol.). Splints. Chlorine-water. Bromine-water.

a. Pour into the evaporating dish 1 c.c. of water and add 2 c.c. of concentrated sulphuric acid. Pulverize finely about 1 c.c. of potassium iodide, add 2 c.c. of manganese dioxide, mix, and add the mixture to the acid. Place the evaporating dish upon the ring on the stand and set the beaker with about 50 c.c. of water upon it so that the vapor of the iodine may condense on the bottom of the beaker.

Heat the mixture gently with a very small flame, which does not touch the dish. Observe the color of the vapor(?), and

the crystals(form?) on the bottom of the beaker. What was the color of the potassium iodide(?), and the form of its crystals? Are its crystals like those of iodine either in form or color? While the crystals are accumulating, proceed with d.

b. Place in as many test-tubes 1 c.c. each of water, alcohol, carbon disulphide, and potassium iodide solution. Scrape some of the crystals of iodine off the bottom of the beaker with a wooden splint, add one crystal to the contents of each of the four test-tubes and shake(?). Is the iodine heavier or lighter than water?

Tabulate the results as follows, giving the solvent(?), degree of solubility(slight, considerable, very great?), and the color of the solution(look through it at a piece of white paper?):

SOLVENT	SOLUBILITY	COLOR OF SOLUTION
Water:		
Etc.:		

The solution in alcohol is the lotion called "tincture of iodine." That in potassium iodide solution is used in testing for starch(Ex. 34 b).

c. To the test-tube containing water and iodine, add an equal volume of carbon disulphide and shake(?).

d. **Test for an Iodide.** Take a crystal of potassium iodide (color?) and dissolve it in 5 c.c. of water(color of solution?). Add 2 c.c. of carbon disulphide and shake(?). Why is not the color of iodine visible?

Holding the tube steady, add now a drop or two of chlorine-water, and observe carefully whether the color appears in the carbon disulphide or in the aqueous layer(?) and what color it is(?). Then shake vigorously and allow the mixture to settle. What substance has been liberated? Write the equation [411](?). To what variety of chemical changes does this one belong?

e. Repeat d, using bromine-water instead of chlorine-water(?).

f. Summarize the properties of iodine: color of solid(?), of vapor(?), density of vapor compared with air and with bromine vapor(?), density of solid compared with water(?), solubilities in four solvents(?).

Compare the affinities of chlorine, bromine, and iodine for potassium(?).

EXERCISE 55.

COMPARISON OF THE HALOGEN COMPOUNDS.

Object: *To learn how a chloride, a bromide, and an iodide react with sulphuric acid, and to observe the properties of the hydrogen compounds of the halogens.*

Apparatus: Mortar. Test-tubes. Glass rod.

Materials: Potassium chloride. Sulphuric acid(conc.). Litmus paper (blue). Ammonium hydroxide(sol.). Potassium bromide. Potassium iodide.

Note: Remember that the hydrogen compounds of the halogens are all colorless.

a. Hydrogen Chloride. Pulverize about 1 g. of potassium chloride(color?), place it in a test-tube, and add 0.5 c.c.(not more) of concentrated sulphuric acid(?).

Blow the breath across the mouth of the tube(?).

Test the gas with moistened blue litmus paper(?).

Lower into it a glass rod dipped in ammonium hydroxide solution, which will give off ammonia gas(?). Write the equation(?).

b. Is any colored gas visible in a? Why is the color of chlorine not observable?

c. Hydrogen Bromide. Repeat a in every detail using potassium bromide, make the same notes(?) and answer the same questions(?).

d. Is there a colored gas or vapor visible in c? Name

it(?). How could you distinguish a bromide from a chloride?

e. **Hydrogen Iodide.** Repeat a in every detail, using potassium iodide, make the same notes(?) and answer the same questions! ?).

f. Warm the tube from e(?). What is the colored vapor? Does it form crystals on the tube? Stop heating, and let the vapor condense. What gas can you recognize by its odor [CAUTION]?

Was this odorous gas formed from the potassium iodide or the sulphuric acid (in answering, consider the formulæ of these substances and of the gas)? By what sort of chemical action must this gas have been formed? Could hydrogen iodide have produced such an action? Write an equation to illustrate your answer(?). Why, then, was iodine liberated?

g. Using the results of this exercise, how could you recognize a bromide and an iodide, and distinguish them from a chloride?

What other method of recognizing bromides and iodides did we learn before?

EXERCISE 56.

PHOSPHINE AND PHOSPHORIC ACID.

Object: To prepare phosphine and to observe the reactions of phosphoric acid or phosphates.

Apparatus: Test-tubes. Glass tubing. Mortar. Dropper.

Materials: Calcium phosphide. Litmus papers. Phosphoric acid (sol.) or sodium phosphate. Ammonium molybdate(sol.*). Silver nitrate(sol.). Ammonium hydroxide(sol.). Nitric acid(conc.).

a. **Phosphine.** Drop a small piece of calcium phosphide Ca_3P_2 , half the size of a pea, into 3 c.c. of water in a test-tube(?). The gas is phosphine PH_3 . Try the reaction of the gas(not the liquid) with moist litmus papers(?). What

* 70 g. ammonium molybdate and 180 c.c. nitric acid to make 1 liter of solution.

reaction would ammonia NH_3 gas give? Note the odor(?). Is the gas combustible? Is its kindling temperature low or high?

Test the reaction of the water(?). What must the dissolved substance be? Write the equation for the action of the calcium phosphide on water(?).

b. Test for the Phosphate Radical. To 10 c.c. of water add not over 1 c.c. of phosphoric acid H_3PO_4 (or 0.5 c.c. of sodium phosphate). Take 5 c.c. of ammonium molybdate solution, add to it 2 drops (not more, reserve the rest for c) of the phosphoric acid solution and warm the mixture(?). This is a test for the radical of orthophosphoric acid (PO_4) and the phosphates.

c. To the rest of the phosphoric acid solution add silver nitrate solution, shaking and adding more until a permanent precipitate is formed(?). The precipitate is silver orthophosphate Ag_3PO_4 . Write the equation(?).

Like all double decompositions, this one is reversible, and is therefore incomplete. Name the interacting substances in the reverse action(?). To stop this reverse action, destroy the acid by adding ammonium hydroxide solution one drop at a time, noting the effect, **shaking**, and examining again. What evidence is there that **the action** now goes further forward?

Finally, add concentrated **nitric acid**(?). Which way does the action now go?

EXERCISE 57.

ARSENIC TRIOXIDE.

Object: To observe the reduction of an oxide by carbon and to study the properties of arsenic trioxide.

Apparatus: Glass tubing. Test-tubes.

Materials: Arsenic trioxide. Charcoal (pulv.). Hydrochloric acid (conc.). Sodium hydroxide (sol.).

a. Draw out a piece of glass tubing to make two ignition tubes each 7 cm. long. Place in the closed end of one a very small amount of arsenic trioxide. Heat the oxide, and watch the cold part of the tube for a sublimate [411](?). Is the oxide volatile? Does it melt before vaporizing?

b. Place a small amount of arsenic trioxide in the other tube, and above it a little powdered charcoal. Heat the oxide and the charcoal, and examine the cold part of the tube [437](?). What change has the oxide undergone?

c. Take about 0.5 c.c. of arsenic trioxide in a test-tube, add 2 c.c. of water and warm(?). Now add 1 c.c. of concentrated hydrochloric acid and heat again(?). To what class of oxides does arsenic trioxide appear to belong [349]? Write the equation(?). Set aside and examine later. Is the action reversible?

d. Take about 0.5 c.c. of arsenic trioxide, add 2-3 c.c. of sodium hydroxide, and warm(?). To what class of oxides does it appear now to belong?

EXERCISE 58.

BORAX BEADS TESTS.

Object: *To learn the use of a borax bead for recognizing the metallic elements in an unknown compound.*

Apparatus: Watch glass. Platinum wire.

Materials: Borax(pulv.). Cobalt chloride. Manganese dioxide. Potassium dichromate. Ferric chloride. Nickel sulphate. Cupric sulphate. Unknowns.

a. Place some powdered borax on a watch glass. Heat the platinum wire and dip the glowing end in the borax. Use the straight wire, without any loop. Now hold the wire in the flame and observe the behavior of the borax(?). What vapor causes it to puff up [454]? The bead must be small, to avoid its dropping off.

Lower the flame until it is so small that it can be sheltered from drafts by the hands. Close the air-holes until a small luminous tip appears in the interior of the flame. This is the reducing portion of the flame(Why?). The blue tip and whole exterior of the flame is the oxidizing part(Why?).

b. Heat the borax bead and touch with it a small particle of a compound of cobalt. Hold the glass rod between the first and second fingers of the right hand, so that when the hands surround the flame, the bead is held in the latter. Hold the bead in the oxidizing flame.

Examine the bead when cold(?). If it is not distinctly colored, pick up another particle of the cobalt compound and heat again. If the bead is opaque, too much of the compound has been taken. Remove the bead and start again.

To remove the bead, heat it red hot and, holding it over the sink or waste-jar, tap the wire with a pencil.

Now hold the bead in the reducing flame, persistently, keeping the flame steady by sheltering it with the hands(?).

Finally, remove the bead and make another. If this shows the cobalt color, remove it and repeat until a colorless bead is obtained.

c. Repeat b, touching a particle of manganese dioxide with the heated bead, and examine in the oxidizing(?) and reducing flames(?). Tabulate the results of b, c, d, e, and f as shown below.

d. Repeat c with a compound of chromium(?).

e. Repeat c with a compound of iron(?).

f. Repeat with compounds of nickel(?) and copper(?).

COMPOUND USED	FORMULA	COLOR OF BEAD	
		OXID: FLAME	RED: FLAME

g. Obtain an unknown substance and identify the metallic element in it.

EXERCISE 59.

SODIUM BICARBONATE. ACID SALTS. BAKING POWDER.

Object: *To prepare sodium bicarbonate by the Solvay process. To study the effect of heat on the bicarbonate and on another acid salt. To observe the behavior of a baking powder.*

Apparatus: Graduated cylinder. Test-tube (large), and cork. Trip scales. Gas generating bottle with thistle, L, rubber, and glass delivery tubes. Test-tube, 1-hole stopper and L-tube. Test-tubes.

Materials: Ammonium hydroxide (sol.). Ammonium carbonate. Sodium chloride. Marble (chips). Hydrochloric acid. Filter-paper. Limewater. Sodium bisulphite. Potassium bitartrate.

a. Preparation. Measure 24 c.c. of ammonium hydroxide solution and 12 c.c. of water into a large test-tube, add 8 g. of powdered ammonium carbonate, cork the tube and shake until the salt is dissolved. Add solid, powdered sodium chloride in excess and shake vigorously until the liquid is saturated.

Decant the clear liquid into another test-tube, and lead into it carbon dioxide (made as in Ex. 30 a) until a copious precipitate of sodium bicarbonate has appeared. While this is going on, proceed with b.

Filter, and dry the precipitate by pressing between filter papers. Note the appearance (?) and taste (?).

b. Effect of Heating the Bicarbonate. Place some sodium bicarbonate in a test-tube so that the mouth is inclined slightly downwards, with the L-tube dipping into limewater. Warm the bicarbonate gently with a small flame. What gas is given off? What is deposited in the cool part of the tube? Taste the residue when cold? To a part, add an acid (?). What is the residue? Write the equation for the action of heat on the bicarbonate (?).

c. Effect of Heating Another Acid Salt. Many acid salts [216] behave, when heated, as sodium bicarbonate did in b, and can be recognized by this behavior. Heat a little sodium bisulphite in a test-tube, clamped horizontally (?). What gas

(odor?) is given off? Is water liberated? Write the equation(?).

d. Summarize the properties of sodium bicarbonate and of sodium carbonate, namely, color(?), taste(?), effect of heating(?).

e. **Baking Powder.** Weigh out 2 g. of potassium bitartrate(cream of tartar). Write the equation for its interaction with sodium bicarbonate [462](?). Calculate how much of the latter salt will be required(?). Weigh out this amount, and mix the two salts on paper. In practice, corn starch is also added(Why?), but is not here required.

Put half the mixture in cold water in a beaker(?). Put the other half in water previously heated(?). Why do soda biscuits rise so rapidly when placed in the oven?

EXERCISE 60.

ANALYSIS OF BAKING POWDER.

(Qualitative.)

Object: *Baking powders contain sodium bicarbonate, a substance which is acid(or becomes so on being heated), and starch to delay interaction. The acid(or acid-forming) substance may be potassium bitartrate $KHC_4H_4O_6$, or calcium acid phosphate $Ca(H_2PO_4)_2$, or potassium bisulphate $KHSO_4$, or ammonium alum $(NH_4)_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$. The object is to learn tests for the presence of the radicals of these substances.*

Apparatus: Test-tubes. Funnel. Evaporating dish. Platinum wire.

Materials: Baking powder. Filter-paper. Sulphuric acid(conc.). Nitric acid(dil.). Ammonium molybdate(sol., Ex. 56). Hydrochloric acid(dil.). Barium chloride(sol.). Sodium hydroxide(sol.). Litmus papers. Ammonium hydroxide(sol.). Acetic acid(dil., 6N). Ammonium oxalate(sol.). Iodine(sol. in KI). Cobalt chloride(sol.).

a. **Preparatory.** Place about 4 c.c. of the baking powder into a large test-tube, add 20 c.c. of water, and shake vigor-

ously for several minutes. Filter the liquid and test the clear filtrate as in c, d, e, and f.

b. **Starch** see Ex. 54 b. Perforate the filter paper and with water wash some of the residue into a test-tube. Boil the suspension, and then cool it and fill the test-tube up with water. With a glass rod add to the suspension one drop of the iodine solution. A blue color indicates starch.

c. **Tartrate Radical.** Place 4 c.c. of the filtrate a' in an evaporating dish with 5 drops of concentrated sulphuric acid and evaporate to dryness over a small flame. Charring and an odor of burnt sugar indicate a tartrate.

d. **Phosphate Radical** see Ex. 56 b. Take 1 c.c. of the filtrate (a), and acidify with 1 c.c. of dilute nitric acid. To 5 c.c. of ammonium molybdate solution add 3 drops of this mixture, warm, and set in the rack. Phosphates give a yellow precipitate.

e. **Sulphate Radical** (see Ex. 46 d). Acidify about 5 c.c. of the filtrate (a), with 5 c.c. of dilute hydrochloric acid and add barium chloride solution (?). What is the precipitate (if any) ?

f. **Ammonium Radical** (Ex. 40 f). To 5 c.c. of the filtrate (a), add 5 c.c. of sodium hydroxide solution. Heat to boiling and note the odor (?) and reaction of the vapor (not the liquid) towards moist red litmus paper (?). What is the gas (if any) ?

g. **Calcium Radical.** Shake about 0.5 c.c. of the baking powder with 5 c.c. of dilute hydrochloric acid. Filter and to the filtrate add ammonium hydroxide until the liquid is alkaline to litmus. Now add acetic acid until the solution is acid to litmus, boil, and filter if the liquid is not clear. Add ammonium oxalate solution (See Ex. 27 a c). The precipitate, if any, is calcium oxalate.

h. **Aluminium Radical.** For test, see Exercise 75 a.

j. **Report.** Summarize, stating which radicals were present and which absent(?).

EXERCISE 61. ✓

HYDROLYSIS.

Object: *To examine several salts in order to find out, by the reaction of the solution, which interact with water and which do not, and to explain the result in each case.*

Apparatus: Test-tubes.

Materials: Litmus papers. Sodium carbonate(sol.). Cupric sulphate(sol.). Potassium iodide(sol.). Aluminium sulphate. Lead nitrate(sol.). Sodium nitrate.

a. Test some sodium carbonate solution with litmus papers(?). Explain [465, 466] the reaction of the solution(?).

b. Repeat a with cupric sulphate solution(?) and explain(?).

c. Repeat a with solutions of potassium iodide, aluminium sulphate, lead nitrate, and sodium nitrate(?). Write the equation for hydrolysis in each case(?). Where there is a reaction towards litmus, explain it(?). If there is none, explain why(?).

EXERCISE 62.

POTASSIUM NITRATE—PREPARATION.

Object: *To prepare potassium nitrate, and to study the influence of solubility on the products obtained in double decomposition.*

Apparatus: Test-tubes. Trip scales. 2 beakers. Graduated cylinder. Glass rod. Watch glass. Lens.

Materials: Potassium chloride. Sodium nitrate.

a. Dissolve 22 g. of potassium chloride in 45 c.c. of boiling water in a test-tube.

Boil 20 c.c. of water in a beaker(set on the wire gauze) and add 25 g. of sodium nitrate. When this has dissolved, add

the boiling solution of potassium chloride and continue heating for a minute or so(?).

Allow the mixture to settle and immediately pour the hot liquid off the crystalline residue into another clean beaker. Examine the two products in **b** and **c**, respectively.

b. Pour a few c.c. of hot water on to the crystalline residue, stir with a rod to wash the residue and drain away the liquid.

Taste the residue(?). Dissolve a part in a very little hot water, pour several drops on a watch glass and examine the crystals, when they appear, with a lens(?). Draw a couple of them(?). What is the substance?

c. When it is cold, examine the liquid that was poured off into the beaker in **a**(?). What form do most of the crystals show? Are there any cubical crystals(lens)? Pour away the liquid. Dissolve the crystals in a very little boiling water and set aside. This is called recrystallization. Examine the final crystals and draw two of them(?).

d. Write the equation for the action(?). Examine the following solubilities(grams, dissolving in 100 c.c. of water at the temperatures given):

	10°	100°		10°	100°
Potassium chloride	32	57	Potassium nitrate	13	246
Sodium nitrate	80	182	Sodium chloride	36	39

The action, being a double decomposition, is reversible and therefore incomplete. All four substances were present in the mixture when the latter was first made. Which is the least soluble of the four salts at 100°? Why was the sodium chloride the substance precipitated in the hot mixture? Why did potassium nitrate remain dissolved? Why did it come out so copiously when the liquid cooled? Why were there few crystals of sodium chloride mixed with it?

EXERCISE 63.

RECOGNITION OF SUBSTANCES II.

(Negative Radicals.)

Object: Same as in Ex. 47, which see.

Apparatus: Test-tubes. Glass rod. Watch glass.

Materials: Unknowns (Appendix, VI). Litmus papers. Filter paper. Sodium hydroxide. Limewater. Splints. Ferrous sulphate (sol.). Sulphuric acid (conc.). Potassium dichromate (sol.). Sulphuric acid (dil.). Ammonium molybdate (sol.).

a. Obtain [Instructor] a single unknown solid substance. This will contain one of the following radicals:

peroxide(O_2)	phosphate(PO_4)
nitrate(NO_3)	bicarbonate(HCO_3)
chloride(Cl)	carbonate(CO_3)
bromide(Br)	bisulphite(HSO_3)
iodide(I)	sulphite(SO_3)

The positive radical may be (NH_4), (K), (Na), or (Ca).

Limit yourself to identifying the negative radical and (NH_4). Record immediately the result of each observation. Record negative results also.

b. **External Examination** [485]. Record the state(?), crystalline form(?), color(?), and odor(?).

c. **Solubility and Reaction of the Solution.** With a few particles, try the solubility in water and, if in doubt, use the method in Ex. 10 f(?). Test the solution on litmus papers (?).

d. **Effect of Heating** [486]. Heat 0.5 c.c. Does the substance melt(?), char(?), or otherwise change(?). Note also gases or vapors(?).

If WATER VAPOR is given off(?), the substance may be a hydrate or an acid salt. To prevent cracking of the tube, remove the water with filter paper.

A **SUBLIMATE** indicates a salt of NH_4 (test, Ex. 40 f).

A **COLORLESS GAS** with an **ODOR**(?) may be ammonia or sulphur dioxide (Ex. 59 c). Inference?

A **COLORLESS GAS** with **NO ODOR**(?). Examine for carbon dioxide (lime-water test), oxygen from a peroxide or nitrate (test), or nitrous oxide [397] (test, Ex. 52 c). Inference?

e. **Effect of Sulphuric Acid** [487]. To 0.5 c.c. add 2 or 3 drops (not more) of concentrated sulphuric acid(?). If necessary, warm gently(?).

A **GAS** (bubbling) which **FUMES** in the breath. If accompanied by a **COLOR**ED vapor, it may be hydrogen bromide with free bromine [407, par. 2], or hydrogen iodide with free iodine [414, par. 2]. Nitric acid vapor, also, may be faintly colored with NO_2 [388]. Inference?

A **GAS** which **FUMES**, but is **NOT C**OLORED may be hydrogen chloride, or nitric acid (test, Ex. 50 d). Inference?

A **GAS** (bubbling) may **NOT FUME** and may **NOT BE C**OLORED. If it has an **ODOR**, it may be sulphur dioxide from a sulphite or bisulphite. The latter would give sulphur dioxide under d also, the former would not. Inference?

A **GAS**, **NOT FUMING**, **NOT C**OLORED, and **ODORLESS** may be oxygen from some oxides or a peroxide (test, Ex. 48 d), or carbon dioxide (test) from a carbonate or bicarbonate. The last would give carbon dioxide under d also, a soluble carbonate of K or Na would not, but calcium carbonate [249, 3] or ammonium carbonate would do so. Inference?

If there is **NO GAS** (no bubbling), the substance may be a phosphate (test, Ex. 56 b). Inference?

f. Test the unknown for the (NH_4) radical (Ex. 40 f).

g. **Report**. State your conclusion, with the reasons therefor(?).

h. Obtain a second unknown and proceed as before(?).

EXERCISE 64.

ESTERS. SOAP.

Object: *To learn the nature of fats (which are esters) by forming and decomposing a simple ester. To make soap and observe its properties.*

Apparatus: Test-tubes. Evaporating dish. Glass rod. Trip scales

Materials: Sodium acetate. Alcohol (95%). Sulphuric acid (conc.).

Amyl alcohol (or fusel oil). Methyl acetate. Litmus papers. Fat

or cottonseed oil. Sodium hydroxide. Hydrochloric acid (dil.).

Sodium hydroxide (sat. sol.). Sodium chloride (sol.).

a. Formation of an Ester. To 1 c.c. of sodium acetate in a test-tube add 2 c.c. of alcohol and 1 c.c. of concentrated sulphuric acid. Agitate for a minute or two, warm very slightly (do not boil!), and note the odor [495] (?). This is a test for acetic acid or an acetate. Write the equation (?), and name each substance (?).

b. Repeat, using amyl alcohol [495] instead of ordinary alcohol (?).

c. Hydrolysis of an Ester. Place 10 c.c. of water in one test-tube and 0.5 c.c. of methyl acetate $\text{CH}_3(\text{CO}_2\text{CH}_3)$ in another. Test each with blue litmus paper (?), then mix and test again [498] (?). If the result is not definite, wait a few minutes and test the mixture once more. Write the equation (?), and name each substance (?).

d. Saponification of an Ester: Soap Making. Mix in a test-tube 5 c.c. of cold, saturated sodium hydroxide solution and 5 c.c. of alcohol, shake, and allow to settle (?). Pour off the upper, alcoholic layer into another test-tube, add to it an equal volume of fat or cotton seed oil, and shake (?). Put the liquid into an evaporating dish on the wire gauze and set fire to the contents. Warm the dish with a very small flame to assist in driving off the alcohol. Stir until the flame goes out, and then stop heating. The pasty mass is soap, mainly

sodium stearate and sodium palmitate [498, 499], mixed with glycerine. Write an equation(?). Rub a little with water in the hands(?).

The alcohol is used simply as a common solvent for the fat and the alkali and is employed in the laboratory experiment to save time.

e. Dissolve the soap(e) in a little warm distilled water and cool. To half of the solution in a test-tube add hydrochloric acid and shake vigorously [500](?).

To show that the precipitate is an acid, withdraw it by means of a glass rod, suspend it in 10 c.c. of water in another test-tube, add a few drops of sodium hydroxide solution and heat until solution takes place. Write the equation(?).

To what class of substances does f show soap to belong?

f. To the other half of the cold soap solution, add sodium chloride solution(?). This is called "salting out" [499], and is a case of coagulating a colloid [503]. To what class of solutions does this show soap solution to belong?

EXERCISE 65.

COLLOIDAL SUSPENSIONS.

Object: To prepare a colloidal suspension, and observe its properties.

Apparatus: Test-tubes. Funnel. Square bottle (20-25 c.c.). Optional: 300 c.c. beaker.

Materials: Arsenic trioxide. Filter-paper. Hydrogen sulphide(sol.). Sodium chloride(sol.). Hydrochloric acid(conc.). Calcium chloride(sol.). Sugar. Rosin. Alcohol (95% or denatured).

Optional: Ferric chloride(sol. 5%). Sodium sulphate(sol.). Sodium carbonate(sol.).

a. **Preparation.** Shake some arsenic trioxide with 25 c.c. of cold water vigorously for several minutes, filter, and to the filtrate add an equal volume of hydrogen sulphide solution. Arsenious sulphide As_2S_3 is formed, but remains in colloidal suspension.

b. Optical Examination. Pour the suspension into a square bottle, and examine it in sunlight, or, better still, by holding it close to an incandescent bulb. Look first through it towards the light(?). Does it appear to be transparent and clear, or no? Now look at it from a position at right angles to the direction of the light, and answer the same question(?). With a microscope, the particles in such solutions can be perceived, individually [501].

c. Coagulation. Divide the solution between 5 clean test-tubes. Keep one, corked, for reference. To one add sodium chloride solution(?), to another dilute hydrochloric acid(?), to the third calcium chloride solution(?), dissolve a little sugar in the fourth(?), and observe them from time to time. Note which coagulates last(?).

The positive ion is here the coagulating agent. How does valence affect coagulating power? Do non-ionized, non-colloidal substances like sugar produce coagulation?

After a day or two, does the arsenious sulphide in the reference test-tube coagulate or settle of its own accord?

d. Colloidal Rosin. Dissolve a single particle of rosin in 1 c.c. of alcohol. Add the solution to a test-tube full of water(?). Examine in the light as in b(?). Cork and keep, to see whether settling takes place(?).

e. Summarize the special properties of colloidal suspensions(?).

f. Colloidal Ferric Hydroxide(optional). Boil 300 c.c. of distilled(or soft) water in a large beaker, and add to it, a few drops at a time, 3 c.c. of ferric chloride solution. The salt is thus somewhat hydrolyzed and contains suspended ferric hydroxide $\text{Fe}(\text{OH})_3$ (Color?).

Examine this suspension in the light as in b(?).

Take 5 test-tubes full of the prepared liquid, keep one for reference, and add to each of the others a very dilute solution of one of the following coagulants: sodium chloride,

sodium sulphate, sodium carbonate, calcium chloride. This colloid is coagulated by the negative ion. Note the time required in each case(?). What is the effect of valence?

EXERCISE 66.

How SOAP CLEANSSES.

Object: *To observe the power of soap solution to produce an emulsion, and to clean a test-tube brush, covered with oil and rust, separating the rust and oil from one another as well as from the brush.*

Apparatus: Test tubes. Beaker (100 c.c.). Flask. Test-tube brush.

Materials: Kerosene. Cottonseed oil. Ivory soap (sol. 1:10, hot). Suspension of ferric oxide (pulv.) in cottonseed oil (6 g. in 100 c.c. for whole class).

a. Place 1 c.c. of kerosene in one test-tube and 1 c.c. of cottonseed oil in another. Add about 10 c.c. of water to each, shake vigorously, and place in the rack(?). Is a permanent emulsion formed?

Now add to each 2 c.c. of soap solution, shake again, and observe as before(?).

b. Boil 200 c.c. of water in a flask. Saturate the test-tube brush with the rust suspended in oil and push it into a test-tube. Add about 15 c.c. of the hot water, and work the brush in the tube(?). Does hot water alone remove the rust and oil?

Remove the brush, pour out the water, place 15 c.c. of hot soap solution in the test-tube, and work the brush in the tube as before(?). Remove the brush, pour the contents of the test-tube into a 100 c.c. beaker, and rinse first the brush and then the test-tube with hot water, catching the rinsing-water in the beaker. Have the oil and rust been removed from the brush?

After a short time, examine the contents of the beaker(?). Where is the oil(?) and in what condition? Where is the rust(?) and is it free from oil?

Explain how soap solution removes grease or oil from a large object or a powder(?).

EXERCISE 67.

CALCIUM OXIDE AND HYDROXIDE.

Object: *To observe the formation of calcium oxide, and the actions of water and of an acid upon it.*

Apparatus: Wire gauze. 2 watch glasses. Glass rod. Evaporating dish. Test-tubes.

Materials: Marble(chips). Litmus paper(red). Quicklime(fresh). Hydrochloric acid(dil.).

a. Select two small chips of marble. Place one on the wire gauze and heat with the full flame for 10 minutes(meanwhile proceed with c and d). Examine it and compare its appearance with that of the unheated fragment(?). Write the equation for the change(?).

b. Place two strips of red litmus paper in two watch glasses. Lay one of the fragments from a on each, and moisten with water carried on a glass rod(?). What has been formed by the action of the water? Write the equation(?). What sort of oxide is calcium oxide [527]?

c. Place a lump of quicklime in the evaporating dish, add a little water(only enough to wet the lower quarter of the lump) and warm gently(?). Write the equation(?).

d. Place about 1 c.c. of powdered fresh quicklime in a test-tube, add dilute hydrochloric acid, shake, and warm if necessary(?). Write the equation(?). What sort of oxide is quicklime?

For comparison, take a chip of marble and cover it with dilute hydrochloric acid(?). Write the equation(?). What is the reason for the difference?

e. Summarize the properties of marble and quicklime: color(?), appearance(?), effect of heating(?), action of water(?), action of an acid(?).

EXERCISE 68.

HARD WATER.

Object: *To learn the means of detecting hardness in water, and two ways of removing temporary hardness.*

Apparatus: Test-tubes. Gas generating apparatus. Graduated cylinder. Optional: Burette, 500 c.c. bottle (tall form) and cork.

Materials: Soap solution (12 g. per l.). Limewater. Magnesium chloride (sol.).

Optional: Soap solution (12 g. Castile soap per l. of 65% alcohol), natural hard water.

a. Soft Water. To about 10 c.c. of distilled water add soap solution drop by drop, shaking between drops. Count the number of drops required before a "permanent" lather or froth is obtained(?). A "permanent" froth is defined as one which persists for three minutes.

Give the name and formula of one typical component of soap [498, par. 3]?

b. Temporary Hardness. Take 5 c.c. of saturated limewater (1.7 g. Ca(OH)_2 per liter) and add an equal volume of distilled water. Pass carbon dioxide (made as in Ex. 30 a) through the half-saturated limewater steadily until two changes(?) have occurred (meanwhile proceed with c). Write two equations, one for each change [531](?). The clear product is water of temporary hardness. What substance is present?

c. Calculation of the Degree of Hardness. What weight (grams) per liter of calcium hydroxide was contained in the diluted limewater? What weight (grams) per liter of CaCO_3 would this give in the final product(?). How many degrees of hardness is this in French units [531]? How many grains per U.S. gallon of 58333 grains?

d. Action of Hardness on Soap. To 2 c.c. (measured) of the product from b add soap solution (noting how much) and

shake until a "permanent" lather is obtained(?). The precipitate is a calcium soap. Write the formula of a typical component of this soap(?). Write the equation and name each substance(?).

e. Removing Temporary Hardness(softening). To 2 c.c. (measured) of the product from **b**, add 1 c.c.(measured) of saturated limewater and shake(?). What is the precipitate [533]? Now add soap solution, noting how much is required to form a "permanent" lather(?). Was the amount required as great as in **d**, or was it nearer the amount used in **a**?

f. Boil 2 c.c.(measured) of the product from **b**(?). What is the precipitate? When the liquid is cold, add soap solution as in **e**, to determine whether the hardness has changed(?).

g. Permanent Hardness. Dissolve a crystal of magnesium chloride in 10 c.c. of water, add soap solution and shake(?). Write the equation(?) and name each substance(?).

h. Test the city water with soap solution(?).

j. To Determine the Weight of Soap Destroyed by Ten Gallons of a Hard Water(Quantitative: Optional). Fill the burette with a solution containing 12 g. of Castile soap per liter of 65 per cent alcohol.* In a 500 c.c. bottle, provided with a cork, place 100 c.c. of the hard water. Run in the soap solution, 0.5 c.c. at a time, shaking vigorously after each addition. If 12 c.c.(or less) of the soap solution is not sufficient, stop at that point, throw out the sample, take 50 c.c. of the hard water diluted with 50 c.c. of distilled water, and start again. With very hard water it may be necessary to use as little as 5 c.c. diluted with 95 c.c. of distilled water. More than 12 c.c. of the alcoholic soap solution must

* This solution approximates closely to that used for determining hardness. If accurately standardized, 1 c.c. of this solution, when added to 100 c.c. of a sample of water of 1° hardness (French, 1 part CaCO_3 per 100000) gives a permanent lather. With 2° hardness, 2 c.c. are required per 100 c.c. of water, and so forth.

not be used, because the alcohol then tends to prevent the formation of the lather and renders the results inaccurate. If only 5 c.c. (for example) of the water is used, then the amount of soap solution that would have been required for 100 c.c. is obtained by multiplying the volume of soap solution employed by 20. Record the results as follows:

Vol. of hard water used	c.c.
Vol. of soap solution employed	c.c.
Vol. of soap solution required for 100 c.c. of the water	x c.c.

If the soap solution was standardized, x is also the degree of hardness in parts per 100000.

Since 1000 g. (1 liter) of the soap solution contained 12 g. of soap, 1 c.c. contained 0.012 g. of soap, and 100 c.c. of the hard water destroyed 0.012 g. of soap (in 1 c.c. of the solution) for each degree of hardness.

Hence 100 g. of the hard water destroyed $x \times 0.012$ g. of soap.

Hence 100 lbs. of the hard water destroyed $x \times 0.012$ lbs. of soap.

Or 1 lb. of the hard water destroyed $x \times 0.00012$ lbs. of soap.

Now 10 gallons of water weigh 83.4 lbs.

Hence 10 gal. of the hard water destroyed $83.4 \times x \times 0.00012$ lbs. of soap.

Weight (lbs.) of soap destroyed by 10 gal. (?).

EXERCISE 69.

FLAME TESTS.

(Positive Radicals.)

Object: To observe the characteristic colors given to the Bunsen flame by the heated vapors of compounds of six metals, and to use them for recognizing six positive radicals.

Apparatus: Test-tube. Iron wire(No. 20), or platinum wire. Cobalt glass. (Optional: Spectroscope.)

Materials: Chlorides of lithium, calcium, strontium, barium, sodium, and potassium. Hydrochloric acid (conc.).

a. On six pieces of paper write the names of the six substances listed above and obtain on each a few particles of the proper substance. Provide 5 c.c. of concentrated hydrochloric acid in a test-tube.

Hold the iron(or platinum) wire in the Bunsen flame. If the flame is colored by it, dip the wire in the acid, and hold it in the flame until the latter is as blue as usual. Clean the wire in this way(*i.e.* by vaporizing impurities) before and after each of the following tests.

b. Heat the tip of the cleaned wire and quickly touch the first named of the substances. Hold the wire with adhering particles in the lower part of the outer blue layer of the flame and note the color of the latter(?).

Repeat with the next three substances, and tabulate the results, giving the name of the substance(?), its formula(?), and the color(?).

c. Using sodium and potassium chlorides, view both flames through a piece of cobalt glass. Which tint of light is absorbed by the glass, and does not reach the eye(?), and which tint passes through and is visible?

Mix the two chlorides intimately, and observe the flame with the eye(?). Which color is visible? Why is the other invisible? Now view the flame of the mixture through cobalt glass(?). Which metal can you recognize thus?

d. (Optional) If a spectroscope is available, the spectra, and particularly that of sodium, should be examined.

e. Obtain [Instructor] an unknown and identify it.

EXERCISE 70.*

COMPOUNDS OF MAGNESIUM.

Object: *To study the preparation of compounds by double decomposition, and (in d and e) to observe the properties of the carbonate and oxide of magnesium.*

Apparatus: Test-tubes. Glass rod.

Materials: Magnesium chloride. Sodium hydroxide(sol.). Sodium carbonate(sol.). Ammonium chloride(sol.). Ammonium hydroxide(sol.). Sodium phosphate(sol.). Magnesium carbonate(pulv.). Limewater. Hydrochloric acid(dil.).

a. Dissolve about 0.5 c.c. of magnesium chloride(properties?) in 10 c.c. of water, and divide the solution into three portions.

To one portion add sodium hydroxide solution(?). Remember that salts, bases, and acids usually interact by double decomposition. On this basis, write the equation(?), and name each substance(?).

b. To the second portion(a) add sodium carbonate solution [543](?). Why does the addition of sodium bicarbonate to table salt containing magnesium chloride prevent the salt from becoming moist in damp weather?

c. **Test.** To the third portion(a) add ammonium chloride solution(not included in equation), then a little ammonium hydroxide and sodium phosphate [704, par. 4](?). Rub the inside of the test-tube with a glass rod and note where the precipitate appears(?).

d. Heat 0.5 c.c. of powdered magnesium carbonate in a test-tube and lower into the gas a rod dipped in limewater(?). Write the equation(?). Use the residue—after strong heating—in e.

e. To the residue from d, when cold, add dilute hydrochloric acid(?). What sort of oxide is magnesium oxide?

* If time presses, part or all of this exercise may be omitted. It simply reviews similar phenomena already studied.

EXERCISE 71.

FILM TESTS.

(Metallic Elements.)

Object: *To recognize compounds of arsenic, antimony, bismuth, zinc, lead, and mercury by liberating the metal and studying its properties; also by turning the metal into its oxide and the oxide into the iodide and the iodide into the sulphide, and studying the properties of the oxide, iodide, and sulphide. Incidentally, to review many reactions.*

Apparatus: Evaporating dish (preferably with glazed exterior). Glass rod.

Materials: I—Arsenic trioxide. Antimony trichloride. Bismuth nitrate. Zinc oxide. Lead nitrate. Mercurous chloride. II—Asbestos (long fibered). Nitric acid (dil.). Phosphorus triiodide.* Ammonium hydroxide (sol.). Ammonium sulphide (sol.). Unknowns.

a. Metallic Films [703]. Obtain on small pieces of paper bearing the names of the materials, a few particles of each of the six substances first mentioned above. Lower the Bunsen flame to 5 cm. height, and reduce the air supply until a luminous tip appears inside the flame. Clean the outside of the evaporating dish and place a little water in it. Take a thin straight fiber of asbestos, dip the point in the water, and touch the arsenic trioxide with the moist end, so as to pick up a few particles.

Hold the evaporating dish in the left hand so that a glazed part of its exterior is close above the luminous tip. At the same moment, with the right hand, insert the point of the asbestos fiber quickly into the luminous tip itself. Place the asbestos fiber beside the arsenious oxide, for use again in c, and examine the dish(?). Is there a metallic film, or only a dust on the dish? Dip a glass rod in dilute nitric acid

* In a very small, stoppered, w.-m. bottle put a small piece of white phosphorus and a little powdered iodine. When the reaction is over, or if the phosphorus catches fire, close the bottle.

(in a test-tube) and moisten the film therewith. Does the film dissolve quickly, slowly, or hardly at all? Tabulate the results as shown below.

Repeat with the other five * substances, cleaning off the dish, and using a fresh asbestos fiber for each.

SUBSTANCE	FORMULA	FILM DUSTY OR	DILUTE
		CONTINUOUS	NITRIC ACID

Arsenic trioxide:

b. What reducing substances are present in the luminous tip? Write the equations for the interaction of carbon with each of the oxides, and of hydrogen with each of the chlorides used. The two nitrates give first the oxides when heated. How is metallic zinc prepared from the oxide commercially [545] ?

c. **Oxide Films.** Lower the flame still further, so as to concentrate the film on a small part of the dish. Repeat the first part of a with the first five substances,** holding each in the reducing tip, but holding the dish higher up in the flame, so that the metal may be re-oxidized and the oxide caught on the dish. Note the color of each oxide(?), tabulate the results as shown under g, and use each oxide for d, e, f, and g, before cleaning the dish and proceeding with the next substance.

d. **Iodide Film.** Take the dish with the oxide film to the bottle containing phosphorus triiodide, and blow across the mouth of the bottle so that the gas strikes the oxide, converting it into the iodide(color?).

e. Breathe upon the iodide film heavily once or twice(?). Soluble iodides become invisible, but reappear on drying.

f.† When the iodide film has reappeared, blow across the

* If the period is brief, three may be handled one day and three the next.

** Mercury gives the metal, and very little of it is oxidized.

† If the period is brief, f or g may be omitted.

mouth of the ammonium hydroxide bottle, so that the ammonia strikes the iodide(?). If the color vanishes, watch whether it reappears by re-escape of the ammonia(?).

g. Blow across the mouth of the ammonium sulphide bottle, so that the hydrogen sulphide strikes the residue on the dish. This converts it into the sulphide(color?).

METAL	COLOR OF		BEHAVIOR OF THE IODIDE WITH		
	OXIDE	IODIDE	MOISTURE	AMMONIA	AM. SULPHIDE
Arsenic:					

h. Obtain an unknown, and identify it.

EXERCISE 72.

ALUMINIUM HYDROXIDE. ALUM.

Object: *To prepare a double salt in beautiful crystals. To observe the formation of aluminium hydroxide, and its properties of coagulating suspended matter and forming lakes with dyes.*

Apparatus: Test-tubes (large). Glass rod. Trip scales.

Materials: Ammonium sulphate. Aluminium sulphate. Thread. Aluminium sulphate(sol.). Litmus papers. Limewater. Ammonium hydroxide. Clay.

a. **Ammonium Alum—a Double Salt.** Calculate what weight of ammonium sulphate $(\text{NH}_4)_2\text{SO}_4$ is required to give as many molecules as are contained in 10 g. of aluminium sulphate [560](?). Weigh out the equivalent quantities of the two salts, and dissolve them separately, each in the smallest possible amount of hot water.

Then mix the clear solutions, suspend a thread tied to a glass rod in the mixture, and set it aside to crystallize. Note the form(?), color(?), and taste(?) of the crystals.

b. **Aluminium Hydroxide.** Dilute 2 c.c. of aluminium sulphate solution with 20 c.c. of water.

To half of the solution add about 15 c.c. of limewater(?). Write the equation.

To the other half add ammonium hydroxide(?). Equation(?).

c. Coagulation. Take 100 c.c. of water with clay in suspension, add 1 c.c. of aluminium sulphate solution, stir vigorously, and then add 15 c.c. of limewater. Observe the water after it has stood for some time(?). Explain [562](?).

d. Lakes [566]. Take some logwood solution, add to it a few drops of ammonium hydroxide, and shake. Then add 1 c.c. of aluminium sulphate solution, shake, and allow to stand(?).

EXERCISE 73.

DYEING I.

Object: *To try two dyes, one of which dyes cotton directly, while the other will dye it only with the help of a mordant.*

Apparatus: Evaporating dish. Graduated cylinder. Test-tubes. Glass plate.

Materials: White cotton cloth (pieces 5 x 2 cm.). White flannel (5 x 2 cm.). Hydrochloric acid (conc.). Ammonium hydroxide. Chrysophenin (suspension, 1%). Sodium sulphate (sol., 8 g. Na_2SO_4 , 10H₂O per l.). Alizarin (suspended, 5 g. of 20% paste to 100 c.c. water). Aluminium sulphate (sol., N).

a. Preparing the Cotton. In case the sizing has not been removed from the cotton cloth, boil seven pieces in 50 c.c. of water containing 2 c.c. of concentrated hydrochloric acid. Rinse the goods in water, dip in 50 c.c. of cold water containing 0.5 c.c. of ammonium hydroxide, and rinse again.

Use three pieces in b and c and keep four for Ex. 74.

b. Chrysophenin, a Direct Dye on Both Cotton and Wool. To 20 c.c. of water in the evaporating dish add 5 c.c. of the chrysophenin suspension (shake the bottle) and 1 c.c. of sodium sulphate solution, and heat to boiling.

Place in this bath one piece each of flannel and of cotton and keep them in motion with a glass rod for two minutes.

Remove them and wash in running water(?). Is the dye fast to washing on both? What was the purpose of the sodium sulphate [566, par. 4]?

Smooth out the samples on a square of glass (or a bottle) to dry, and paste them in your note book.

c. **Alizarin, a Non-basic, Mordant Dye.** In a test-tube dilute 2 c.c. of aluminium sulphate solution with 10 c.c. of water, place in it a piece of cotton cloth and boil for two minutes.

In a second test-tube dilute 1 c.c. of ammonium hydroxide with 10 c.c. of water. Wring the piece of cloth, place it in this solution and warm and shake for two minutes. Then wring the cloth, which is now mordanted with aluminium hydroxide.

In the evaporating dish put 50 c.c. of water and 5 c.c. of the alizarin suspension (shake the bottle). Place in this the piece of mordanted cloth and a piece of unmordanted cotton, and heat to boiling. Keep the pieces of cloth in motion for at least ten minutes. Finally, wash them in running water(?). Is the dye fast to washing on both? Why do they differ [566, par. 3]?

Dry the samples on glass and paste them into the note book.

EXERCISE 74.

DYEING II.

Object: *To try two dyes, using a different mordant.*

Apparatus: Test-tubes (large). Graduated cylinder. Evaporating dish. Glass plate.

Materials: Malachite Green (sol. 0.05%). Methyl violet (sol., 0.05%). Tannic acid (sol., 1%). Tartar emetic (sol., 1%). Labels. Four pieces cotton (from Ex. 73 a).

a. **Basic, Mordant Dyes.** Measure into as many large test-tubes the following solutions:

- (1) 2 c.c. Malachite Green (3) 2 c.c. Tannic Acid
(2) 2 c.c. Methyl Violet (4) 2 c.c. Tartar Emetic

Add to each 20 c.c. of distilled water, and label and number the tubes.

b. Place two pieces of cotton cloth (prepared in Ex. 73 a) in the evaporating dish, add the contents of (3), and keep at the boiling point for five minutes. Wring the pieces of cloth and place them in (4), which is cold, and keep them in motion for five minutes. Then remove and wring the cloth, which is now mordanted with antimonyl tannate [566, par. 3].

c. **The Dye without Mordant.** Place in (1) and in (2) one piece each of unmordanted cloth, and keep each solution at the boiling point for one minute. Wash the samples thoroughly in running water (?). Is the dye fast to washing?

d. **The Dye with Mordant.** Now place in (1) and in (2) one piece each of the mordanted cotton from b, boil each for one minute, allow them to cool, and then wash the cloth thoroughly (?). Is the dye fast to washing? Dry all the samples on glass and mount in the note book.

EXERCISE 75.

COBALT CHLORIDE TESTS.

(*Metallic Elements.*)

Object: *To learn a test which works well for aluminium and for zinc [see 547, pars. 3 and 4].*

Apparatus: Iron or platinum wire (or blowpipe and plaster or charcoal block). Glass rod.

Materials: Aluminium sulphate. Filter paper. Cobalt chloride (sol.). Zinc oxide. Magnesium carbonate. Unknowns.

a. **Aluminium.** Wrap a few particles of a compound of aluminium, such as aluminium sulphate, tightly in a small piece of filter paper, and wind the platinum wire spirally

round the mass. Char the little ball in the Bunsen flame, moisten it with cobalt chloride solution, applied with a glass rod, and heat again(?). The color(?) of the ash [558] is characteristic.

(Alternative.) If a blowpipe is available, the compound may be placed in a hollow on a block of plaster of Paris or of charcoal, and the heating be done with the tip of the flame.

b. **Zinc.** Repeat a, using any compound of zinc, such as zinc oxide [547].

c. **Magnesium.** Repeat a, using any compound of magnesium, such as magnesium carbonate or sulphate [704, par. 4].

d. Obtain [Instructor] an unknown and identify it.

EXERCISE 76.

DESTRUCTIVE DISTILLATION OF WOOD AND COAL.

Object: *To study the products obtained by distilling wood and coal, such as charcoal, coke, illuminating gas, etc.*

Apparatus: Test-tube (hard glass), tubing, large test-tubes and beaker. Glass rod.

Materials: Sawdust and wood chips. Litmus papers. Bituminous coal (crushed). Lead nitrate (sol.). Filter paper.

a. **Wood.** Take three-fourths of a hard-glass test-tube full of sawdust and wood chips. Arrange the apparatus as in Fig. 18. The test-tube is inclined slightly downwards towards the mouth. The wide test-tube, in which the distillate is to be caught, is surrounded by cold water, and a nozzle is inserted in one of the holes in the stopper.

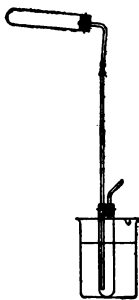


FIG. 18

Heat the contents of the test-tube, at first gently, and later strongly, until no more vapors are evolved.

b. During the heating, set fire to the issuing gas(?). Is

the flame luminous, or not? Compounds of what element must be present?

c. Examine the condensed liquid. What liquid or liquids are seen? Test the liquid with litmus papers(?). What dissolved substance causes this reaction [Wood, 573: Coal, 577]?

d. Examine, describe, and name the residue in the hard glass test-tube(?). Place it in a corked test-tube for use in Ex. 77.

e. Name five observed products from distilling wood(?).

f. **Coal.** Charge a hard glass test-tube with crushed soft coal, attach a clean test-tube to catch the distillate, and repeat a.

g. To test for hydrogen sulphide in the coal gas, dip a glass rod in lead nitrate solution, wipe it on a small piece of filter paper, and hold the latter in the unlighted gas(?). What compound is formed on the paper [344 and Ex. 48 c].

h. Repeat b and c.

j. Examine and name the residue(?).

k. Name six observed products from distilling coal(?).

EXERCISE 77.

WOOD CHARCOAL AND BONE BLACK.

Object: *To study the properties of wood charcoal and of bone black.*

Apparatus: Iron wire. Test-tubes. Crucible (porc.).

Materials: Charcoal (splinters). Splints. Litmus(sol.). Wood charcoal (puly.). Bone black. Molasses. Cupric sulphate(sol.). Ferrous sulphide. Sulphuric acid(dil.).

a. **Charcoal.** Light first a splinter of wood and then a splinter of charcoal in the flame(?). Describe how each burns(?). What is the cause of the flame in one case(?), and of the absence of flame in the other(?).

b. Place a splinter of charcoal in a test-tube half-filled with water. Is charcoal heavier or lighter than water?

Hold the splinter of charcoal under the water by means of an iron wire, one end of which is twisted round it, and boil the water for five minutes. Remove the wire from the splinter. Does the latter now sink or float? Is carbon specifically lighter or heavier than water? Why is fresh charcoal lighter [574]?

c. To a test-tube half full of water add two or three drops of litmus solution. Add about 4 c.c. of powdered charcoal, boil for five minutes, and filter. Where is the litmus? What class of substances, especially, is adsorbed by charcoal from solutions [574]?

d. **Bone Black** [592]. Heat about 15 c.c. of bone black in the crucible (covered). The purpose of the heating is to make the charcoal more active by driving out gases and moisture already adsorbed on the surfaces of its pores.

Take two test-tubes half filled with water, and add to one enough molasses to confer a distinct tint, and to the other a few drops of cupric sulphate solution.

When the bone black has cooled, put about 4 c.c. of it in each test-tube, and shake vigorously.

Filter the dilute molasses. If the filtrate is still colored(?), pour it through the filter again. Taste the filtrate(?). Filter the dilute cupric sulphate, and examine the filtrate(?).

What difference do you observe? Was the sugar removed, or only the coloring matter? Explain(?). What commercial use is made of this property of bone black [287]?

e. Place in a test-tube a small piece of ferrous sulphide, 10 c.c. of water, and 5 c.c. of dilute sulphuric acid. Note the odor(?) and name the gas(?). When the action is well started, decant the liquid to a clean test-tube, add the rest of the heated bone charcoal (from d), shake vigorously, and note the odor(?). What property is here illustrated [574]?

EXERCISE 78.

TESTS FOR FOOD COMPONENTS.

Object: *To learn some tests for starch, glucose, milk-sugar, proteins, and fats, and to apply them to two common foods. Incidentally, to show that starch is partly digested by saliva during mastication.*

Apparatus: Test-tubes. Evaporating dish.

Materials: Starch. Iodine (sol. in KI). Glucose (solid). Fehling's solution (Nos. 1 and II). Milk-sugar. Egg albumen. Nitric acid (conc.). Ammonium hydroxide. Woolen yarn. Fat. Cottonseed oil. Almonds (crushed). Meat. Sausage. Carbon tetrachloride.

a. Starch. Place water in a test-tube, add a pinch of starch, shake, and boil (?). How does the starch change? Cool the suspension in running water, and when it is cold (not before), divide it into two parts and to one add a drop of the solution of iodine in potassium iodide (?). Keep the other half for use in e.

b. Take another pinch of dry starch, and add to it a few drops of the iodine solution (?). Tabulate the results of a and b, as well as those of c, d, f, and g, as follows:

FOOD COMPONENT	REAGENT OR TREATMENT	RESULT
Starch (suspension)	Iodine sol.	?
Starch (grains)	Iodine sol.	?

c. Glucose. Shake about 0.5 c.c. of glucose in 5 c.c. of water. Add 5 c.c. of Fehling's solution (Ex. 34, footnote) and boil (?).

d. Lactose (milk-sugar): Repeat c with about 0.5 c.c. of milk-sugar (?).

e. Maltose from Starch. To 5 c.c. of the starch suspension prepared in a (which itself gives no reaction with Fehling's solution, Ex. 34 a), add about 1 c.c. of saliva, mix, and set aside for fifteen minutes. Then add 5 c.c. of

Fehling's solution and boil(?). How was the maltose produced [586] ?

f. Protein. To a few particles of egg albumen, add a few drops of concentrated nitric acid(?). Add some water to wash off the acid, and pour away the liquid, leaving the solid residue. To the latter add a few drops of ammonium hydroxide(?).

Repeat with a scrap of woolen yarn(?).

Account for the effect of nitric acid on the skin and nails(?).

g. Fat. Place on small pieces of unglazed paper(1) a particle of fat,(2) a drop of cotton seed oil, and(3) part of a crushed almond, put them in the evaporating dish, and warm gently until the fat melts. Examine the papers(?).

h. Examination of a Food. Rub a small piece of raw meat in the mortar with a little water until the color is removed. Then apply to portions of the meat the tests in **a** for starch(?), in **f** for protein(?), and in **g** for fat(?).

j. Take a small piece of sausage and apply to portions of it the tests in **a**, **f**, and **g**. If the results of **a** and **f** are not definite, dissolve away the fat by shaking the fragments of sausage with carbon tetrachloride, and try **a** and **f** again with the residue(?).

EXERCISE 79.

FOOD COMPONENTS OF MILK.

Object: *To find the food components present in milk. To explain curdling. To learn a test for a common preservative, formaldehyde.*

Apparatus: Evaporating dish. Test-tubes. Glass rod.

Materials: Milk. Nitric acid(conc.). Ammonium hydroxide. Acetic acid. Fehling's solution(Nos. I and II). Litmus papers. Iodine(sol. in KI). Formaldehyde(1% sol.). Sulphuric acid(conc.). Ferric chloride(sol.).

a. Heat 50 c.c. of milk to boiling in the evaporating dish.

With a glass rod, fish out the skin which forms on the surface and transfer it to a test-tube. Apply to it the test in Ex. 78 f for protein(?). Name the substance [585]?

To the milk add three or four drops of acetic acid and stir(?). How is this result to be classified? What sort of substances cause milk to curdle? What is the connection with "sour milk"? Filter the acidified milk and test the residue on the filter for protein(?).

b. To 5 c.c. of the filtrate add 5 c.c. of Fehling's solution, and boil(?). What is shown to be present(Ex. 78 d)?

c. To 5 c.c. of the filtrate from a add iodine solution(?). For what food component is this a test? Is it present?

d. Put a few drops of unboiled milk on a piece of unglazed paper and heat as in Ex. 78 g(?). Result?

e. Which food components have you found in milk?

f. **Preservative—Formaldehyde.** To 5 c.c. of milk in a test-tube add an equal volume of water and, by means of a glass rod, one drop(not more) of formaldehyde solution and shake. Take 5 c.c. of concentrated sulphuric acid and add to it by a glass rod one drop of ferric chloride solution. Now hold the test-tube of milk almost, but not quite vertical, and pour the sulphuric acid in a continuous stream down the side of the tube so that it may go to the bottom and form a layer under the milk. What color appears where the liquids meet? Repeat with diluted milk free from formaldehyde(?).

EXERCISE 80.

FOOD COMPONENTS OF FLOUR.

Object: To find the food components in wheat flour.

Apparatus: Evaporating dish. Test-tubes. Beaker.

Materials: Flour. Cheese cloth(squares). Thread. Iodine(sol. in KI). Fehling's solution(Nos. I and II). Nitric acid(conc.). Ammonium hydroxide.

a. Take about 10 c.c. of flour, make it into a dough with water, and then place it in a small piece of cheese cloth. Bring the corners together and with a thread tie the cloth so as to enclose the dough in a bag.

Knead the bag in water in the evaporating dish so long as the liquid squeezed out appears to be more milky than that in the dish. Then pour the milky liquid into a beaker to settle.

Open out the cloth and wash the contents in running water until the wash-water is no longer milky.

b. Test portions of the residue in the cloth for starch(?), glucose(?), and protein(?), and record the results(?).

c. Test a pinch of the dry flour for fat(?).

d. Pour the water away from the sediment in the beaker (from a) and test portions of the solid for starch(?), glucose(?), and protein(?), and record the results(?).

e. Summarize the food components found in flour(?).

EXERCISE 81.

REACTIONS OF IRON COMPOUNDS.*

Object: *To try the tests for ferrous and ferric salts, and to learn how ferrous salts are changed into ferric salts by oxidation and vice versa.*

Apparatus: Test-tubes. 1-hole stopper. L- and delivery tubes. Funnel.

Materials: Ferrous sulphate. Ferric chloride(sol.). Potassium ferrocyanide(sol.), ferricyanide(sol.), and sulphocyanate(sol.). Ferrous chloride(sol.). Chlorine-water. Ammonium hydroxide. Ferrous sulphide. Hydrochloric acid(dil.). Iron(pulv.). Filter paper.

a. **Tests for Ferrous and Ferric Salts.** Wash some crystals of ferrous sulphate in water until the surface layer has been

* If necessary, shorten the exercise by assigning b and d to one set of pupils and c and e to another.

removed by solution. Dissolve them in 15 c.c. of water and divide into three parts.

Dilute 5 c.c. of ferric chloride solution with 10 c.c. of water and divide into three parts.

Test each of the salts with the three following reagents and tabulate the results:

POTASSIUM FERROCYANIDE	POTASSIUM FERRICYANIDE	POTASSIUM SULPHOCYANATE
---------------------------	---------------------------	----------------------------

Ferrous:

Ferric:

In the table, note the color produced(?), and whether there is a precipitate or simply a colored solution(?).

Write the equations for the interactions with the ferric salts [613, 614](?). Potassium sulphocyanate is $K(CNS)$.

Give one distinctive test for a ferrous salt(?) and two for a ferric salt(?).

b. Oxidation of Ferrous Compounds. To a solution of ferrous chloride add an oxidizing agent [609] such as chlorine-water, hydrogen peroxide(acidified), or bromine water. Then test for ferric-ion, using tests from **a**. What change has the ferrous-ion undergone?

c. Boil some water in a test-tube to remove dissolved oxygen, and cool in running water. Wash off the surface layer from a crystal of ferrous sulphate, and dissolve it in the boiled water. Now add a few drops of ammonium hydroxide(?). Shake the mixture with air, and note any changes(?). Give the name and the formula of the first precipitate(?) and of the final product(?).

d. Reduction of a Ferric Compound. Dilute some ferric chloride solution. Through half of it bubble some hydrogen sulphide, made(Ex. 43 a) in a test-tube provided with a 1-hole stopper and delivery tube(?). Test small portions until the liquid no longer gives the reactions for a ferric salt.

The precipitate is sulphur. What change has the ferric-ion undergone?

e. Boil the other half of the ferric chloride solution with iron powder for several minutes, filter, and test quickly for ferrous-ion(?). Write the equation [509].

EXERCISE 82.

COMPOUNDS OF LEAD. WHITE LEAD.

Object: To make white lead, and to learn the properties of three other compounds of lead.

Apparatus: Test-tubes. 90 c.c. bottle, 2-hole stopper, L-, thistle, and delivery tubes.

Materials: Acetic acid (dil. 6N). Lead monoxide. Hydrochloric acid (dil.). Marble. Lead nitrate(sol.). Potassium chromate(sol.). Potassium iodide(sol.).

a. In a test-tube, take 5 c.c. of acetic acid, and on a piece of paper about 3 c.c. of lead monoxide. Boil the acid, and add the lead oxide a very little at a time, noting what becomes of it(?). Finally add more water, boil, and filter to get rid of any unused lead oxide.

Since the oxide interacts with acids, to what class of oxides does it belong [527]? What substance is here formed [631]?

b. Generate carbon dioxide(Ex. 30 a) and lead the gas through the filtrate(?). Give the name and formula of the precipitate [632, par. 3](?). What industry uses this process?

c. Dilute 5 c.c. of lead nitrate solution and divide it into three parts. To one add dilute hydrochloric acid(?). Write the equation and name each substance(?). Indicate which is the insoluble one [633](?).

d. To the second add potassium chromate solution(?) and answer the same questions as in c.

e. To the third add potassium iodide solution(?) and an-

swer the same question as in c. Boil the mixture, allow it to settle, pour off the liquid into a clean test-tube and observe it as it cools(?). Explain(?).

EXERCISE 83.

SEPARATION OF LEAD, MERCURY AND SILVER.

Object: *To learn how to use the properties of the compounds of lead, mercury, and silver for the purpose of separating these elements from a mixture and identifying each when separated.*

Apparatus: Test-tubes. Funnel.

Materials: Lead nitrate(sol.). Hydrochloric acid(dil.). Potassium chromate(sol.). Mercurous nitrate(sol.). Ammonium hydroxide. Silver nitrate(sol.). Filter-paper. Nitric acid(conc.). Litmus papers. Hydrochloric acid(conc.). Copper foil (5 x 50 mm.). Unknowns.

a. Lead Chloride. Dilute 2 c.c. of lead nitrate solution with 5 c.c. of water and add dilute hydrochloric acid, drop by drop, shaking between drops and allowing the precipitate (name it?) to settle, until the next drop produces no further precipitation.

b. When the precipitate has settled, pour away the liquid (what does this contain?) Shake the precipitate with a little distilled water, and allow it to settle, and again pour off the liquid. Now boil the precipitate with distilled water(?).

c. Divide the solution into two parts. Set one aside for later observation(?). To the other add potassium chromate solution(?). How should you recognize a soluble salt of lead?

d. Mercurous Chloride. Repeat a and b, using 2 c.c. of mercurous nitrate solution(?). Does the precipitate(name it?) dissolve on boiling?

Add to the suspended precipitate some ammonium hydroxide [659, par. 3](?). How should you recognize a soluble mercurous salt?

e. **Silver Chloride.** Repeat a and b, using 2 c.c. of silver nitrate solution(?). Does the precipitate dissolve on boiling?

Divide the suspended precipitate into two parts. Set one in the strongest light available and examine it later(?).

To the other part add ammonium hydroxide [667](?).

f. **The Separation.** Mix in one test-tube 2 c.c. each of solutions of lead nitrate, mercurous nitrate, and silver nitrate. Dilute with water and add dilute hydrochloric acid until the precipitation is complete. Filter, and wash the precipitate with a little cold water.

To separate the lead, pour 50 c.c. of boiling water through the filter, taking care to pour it over every part of the precipitate. Which compound will dissolve? Test the filtrate for the presence of this compound(?).

Next, to separate the silver, dilute 5 c.c. of ammonium hydroxide with water, place a clean vessel under the funnel, and pour the solution over the precipitate(?). What do you observe? Which compound is now dissolved(?) and which remains upon the filter?

To the filtrate, add concentrated nitric acid, until the ammonium hydroxide is neutralized(test on litmus papers)(?). Name the precipitate, and summarize the properties by which it may be recognized: Color(?), crystalline or amorphous(?), behavior towards ammonium hydroxide(?), towards light(?).

Finally, to get the residue on the filter into solution for the purpose of testing it for mercury, make a little *aqua regia* by adding 1 c.c. of concentrated hydrochloric acid to 0.5 c.c. of nitric acid, warm the mixture, and pour it on to the residue. Catch the filtrate in a clean test-tube. Dilute the filtrate with 5 c.c. of water and put into it a small strip of clean copper foil. After a few minutes, wash the foil, rub it gently, and examine [659 par. 4](?).

g. Obtain an unknown mixture [Instructor] and separate and identify the metals of this group contained in it. Re-

member that negative results must be reported as well as positive ones.

EXERCISE 84.

DISPLACEMENT OF METALS. COUPLES.

Object: *To study the displacement of metal-ions and hydrogen-ion by three metals. Also in f to observe the effect of a foreign metal on the displacement of hydrogen from an acid by zinc, and thereby to explain the qualities and defects of galvanized iron and tin plate.*

Apparatus: Test-tubes.

Materials: Copper foil. Zinc, sheet. Lead, sheet. Sand paper. Copper wire (No. 30). Labels. Lead nitrate(sol.). Mercurous nitrate(sol.). Silver nitrate(sol.). Cupric sulphate(sol.). Sulphuric acid(dil.).

a. Clean the metals with sand paper, and prepare five strips (5×30 mm.) of copper foil, six strips of sheet zinc and three strips of sheet lead. Attach to each, with the exception of one piece of zinc (reserved for f), a piece of copper wire 20 cm. long.

b. **Zinc.** In five test-tubes take 5 c.c. each of solutions of the nitrates of lead, mercury and silver and of cupric sulphate and dilute sulphuric acid. Label them, noting on the label the name, formula, and positive ion in each case. Place a strip of zinc half-way into each solution and examine from time to time(?). Tabulate the results of b, c, and d as shown below. Keep the strips of zinc for use again in f.

c. **Lead.** In three test-tubes take 5 c.c. each of solutions of mercurous nitrate, silver nitrate and cupric sulphate and label them as in b. Place a strip of lead halfway into each solution and examine later(?).

d. **Copper.** Repeat b, using 5 strips of copper(?). Record negative as well as positive results.

METAL IN STRIP	SOLUTION TAKEN SUBSTANCE	ELEMENT POS. ION	LIBERATED	SOLUTION FORMED SUBSTANCE	POS. ION
Zinc	Lead nitrate	Pb ⁺⁺	Lead	Zinc nitrate	Zn ⁺⁺
Etc.					

e. For each positive result write an ordinary equation, and also an ionic equation [633, par. 2] (?). When the result is negative, explain why it is so [63] (?).

f. **A Couple.** Place 5 c.c. of dilute sulphuric acid in each of five test-tubes. Take the sixth strip of zinc (with no wire) and the four strips of zinc which in b have been immersed in the salts (not the one placed in the acid), and immerse each of them as completely as possible in a portion of sulphuric acid (?). Do you observe any difference in the behavior of the clean strip and those partly coated with metal? Those coated with a foreign metal are called "couples." Which metal of the couple is attacked—the more active or the less active one?

Galvanized iron [546] and tinplate [638] are couples. Name the pair of metals in each (?). When each is corroded by the chemical action of water or acids, which of the two metals in each case goes into combination? In your everyday experience, which is more apt to rust, galvanized iron or tinned iron (tin plate)? Give the reason for what you have observed (?).

EXERCISE 85.

MATCH TESTS.

(Metallic Elements.)

Object: To recognize compounds of copper, tin, silver, lead, iron, cobalt, and nickel by liberating the metal and studying its physical and chemical properties. Incidentally, to review a number of reactions.

Apparatus: Mortar. Watch glass. Magnet (1:10). Platinum wire.

Materials: I—Cupric sulphate. Stannic oxide. Silver nitrate. Lead nitrate. Ferrous sulphate. Cobalt chloride. Nickel sulphate. II—Matches with large sticks. Washing soda crystals. Sodium carbonate(anhydrous). Filter paper. Nitric acid(conc.). Ammonium hydroxide. Hydrochloric acid(conc.). Sulphuric acid(conc.). Potassium chromate(sol.). Potassium ferro-, ferri- and sulpho-cyanides(sols.). Borax.

a. [701, 702]. Obtain, on small pieces of paper bearing the names of the materials, a few particles of each of the seven substances first mentioned above.

Lower the Bunsen flame to 4-5 cm. height and reduce the air supply until a luminous tip appears inside the flame. Cut the head off a match, warm one side of a large crystal of washing soda (hydrated sodium carbonate) in the flame, and rub three-fourths of the match-stick with the melted hydrate. Then hold this end of the match in the flame so as to char it. The purpose is to get a thin piece of charcoal saturated with sodium carbonate.

Place a few particles of one of the substances in the palm of the left hand along with two or three times as much anhydrous sodium carbonate. Add a drop of water and with a penknife blade work the mixture into a stiff paste.

Stick a small mass of the mixture on the charred end of the match, and hold it in the luminous tip until the mass fuses.

Break off the charred end of the match in the mortar, add a little water, and grind the material with strong pressure. Wash away the charcoal and lighter particles, and examine the remaining heavy particles in a good light, turning the mortar so as to cause them to move about.

b. **Copper, Tin, Silver, Lead.** These metals, being malleable, will show minute, flat shining discs(?).

If they are colored(?), transfer some of them with the knife to a piece of filter paper. Touch them with a drop of concentrated nitric acid applied with a glass rod and warm *cautiously* above the flame(?). A green stain indicates cop-

per. Touch now with a drop of ammonium hydroxide(?). A strong blue color [647, par. 2] confirms this conclusion.

If the discs are silver white, transfer some of them to a watch glass, add a drop of concentrated nitric acid, and warm gently above the flame (do not evaporate the acid). A white, insoluble substance indicates tin [637](?). If the discs dissolve, giving a clear solution, add a drop of hydrochloric acid. A white precipitate indicates silver. Confirm (Ex. 83 e)(?). Confirm further by using as in Ex. 83 e a solution of the original substance. If there is no precipitate, add a drop of sulphuric acid. A white precipitate indicates lead [633]. Confirm by treating some of the original substance as in Ex. 83, abc.

c. **Iron, Cobalt, Nickel.** These metals give only heavy, grey particles. Magnetize the penknife by stroking the blade in one direction with one pole of a magnet. Ascertain whether the blade will now attract and hold the particles. To make sure that they are not simply held by the water, wipe them off on a piece of filter-paper, allow them to dry, and try whether the blade now attracts them(?). If it does so easily, they are iron.

Whether the particles were attracted or not, transfer some of them to a piece of filter paper. Moisten them with a drop of hydrochloric acid and warm above the flame(?). A yellow stain indicates iron [609](?). Confirm by one of the tests for a ferric salt (Ex. 81 a). Also apply the tests in Ex. 81 a to a solution of the original substance. A blue stain (while warm) indicates cobalt [621](?). Confirm by applying the borax bead test to the original substance [621]. If there was no distinct stain, add a drop of nitric acid and warm. A greenish stain indicates nickel [624](?). Confirm by the borax bead test with the original substance [624](?).

d. Repeat with each of the other six substances(?).

e. In the case of each of the seven substances taken, make

equations, attaching the name to each formula, as follows:

- (1) Effect of heating with sodium carbonate [701](?).
- (2) Effect of heating the product from (1) [701](?).
- (3) Effect of heating the product from (2) with charcoal [701](?).
- (4) Interaction with reagents applied in b or c. Sources of necessary information are indicated by references in connection with each test.

f. Obtain [Instructor] an unknown and identify it. Make a careful report, showing negative as well as positive results (?).

EXERCISE 86.

MANGANESE AND CHROMIUM.

Object: *To prepare a manganate and a chromate. Also to study the relations of chromates, dichromates, and chromic salts.*

Apparatus: Platinum wire. Test-tubes.

Materials: Manganese dioxide(pulv.). Sodium carbonate(anhydrous). Sodium nitrate. Chromic oxide. Potassium chromate(sol.). Sulphuric acid(conc.). Potassium hydroxide(sol.). Litmus papers. Potassium dichromate(sol.). Alcohol(95%).

a. **Sodium Manganate.** On a minute scale, this substance may be made quickly in the form of a bead. Mix on a piece of paper a few particles each of manganese dioxide, sodium carbonate, and sodium nitrate. Bend the end of the platinum wire into a long, narrow loop. Heat it and touch the mixture. Melt the mass to a bead and heat it in the oxidizing flame(?). Color(?). Write the equation [681](?).

b. **Sodium Permanganate.** Place the wire, with the hot bead, in a narrow test-tube containing some water and, when the bead has dissolved, examine the color of the solution by looking down through it at a piece of paper(?). Now, blow through a glass tube so that the breath(as a source of carbon

dioxide) bubbles through the solution, and continue until a change is observed(?). Write the equation [682](?).

c. **Sodium Chromate.** Repeat a, using chromic oxide in place of manganese dioxide. Color(?). Equation(?).

d. **Potassium Chromate made into Dichromate.** To some potassium chromate solution add a little concentrated sulphuric acid drop by drop and shake [685](?). Color(?). Write the equation and attach the name to each formula(?). What is the anhydride(?), and what the valence of chromium in each of the compounds [387]?

e. **Potassium Dichromate made into Chromate.** To the product from d add potassium hydroxide solution drop by drop until the liquid is faintly alkaline(test on litmus paper). Color(?). Write the equation and attach the name to each formula(?).

f. **Chromic Sulphate from Potassium Dichromate [688].** Alcohol may be used here as the reducing agent. To 5 c.c. of potassium dichromate solution add 10 drops of concentrated sulphuric acid, and 5 drops of alcohol. Warm the mixture(?). The strong green color is due to decomposition of a part of the chromic sulphate(which is purple). When the correct proportions are taken, purple crystals of chrome alum can be obtained from the liquid. The odor(?) is that of aldehyde(CH_3COH), produced by oxidation of the alcohol.

What is the valence [387] of chromium in chromic sulphate [688]?

EXERCISE 87.

RECOGNITION OF SUBSTANCES III.

(*Metallic Elements.*)

Object: To use the properties studied in previous exercises for the purpose of identifying the metallic element in an unknown compound.

130 **RECOGNITION OF SUBSTANCES III** [Ex. 87]

Apparatus: Test-tubes. Mortar. Watch glass. Platinum wire. Evaporating dish. Glass rod. Cobalt glass.

Materials. Unknowns (Appendix, VI). List II, Ex. 85. List II, Ex. 71. Cobalt chloride(sol.). Borax. Solutions of ammonium chloride, carbonate, and hydroxide. Sodium phosphate(sol.). Also for j, splints, limewater, and solutions of sodium hydroxide, barium chloride, ferrous sulphate, potassium dichromate and ammonium molybdate.

a. Obtain [Instructor] an unknown solid substance. The metallic element to be identified will be one of the following:

Aluminium	Chromium	Manganese	Sodium
Antimony	Cobalt	Mercury	Tin
Arsenic	Copper	Nickel	Zinc
Bismuth	Lead	Potassium	
Calcium	Magnesium	Silver	

Record at the time the result of each observation, whether that result is positive or negative.

b. **External Examination** [699]. Record the state(?), color(?), luster, if any(?), crystalline form(?), odor(?).

c. **Solubility and Reaction of the Solution** [700]. Repeat Ex. 63 c(?).

d. **Match Test** [701, 702]. Follow the directions in Ex. 85. If copper, iron, nickel, or cobalt is found, confirm(or reject) by trying the borax bead test(Ex. 58), and compare the results with those tabulated in your notes on that exercise(?).

If lead is found, confirm(or reject) by trying the film tests (Ex. 71) and comparing the results with your notes on that exercise(?).

e. **Film Tests** [703]. In case the results of d are negative, follow the directions in Ex. 71. Compare the results with those tabulated in your notes of that exercise(?).

In case zinc is found, confirm by trying the cobalt chloride test as in Ex. 75(?). In case mercury is suspected, confirm

by heating a little of the dry substance in a test-tube [659, par. 1] (?), and by using the test in Ex. 83 f, par. 5 (?).

f. Cobalt Chloride and Bead Tests [704]. In case the results of **e** are negative, try the cobalt chloride test as in Ex. 75 (?).

If the result is again negative, or if only magnesium is suspected, try the borax bead tests as in Ex. 58. Compare the results with those tabulated in your notes on that exercise(?).

g. Flame Tests [704]. In case the result is still negative, try the flame tests as in Ex. 69(?). If calcium is suspected, confirm by dissolving a little of the original substance and adding ammonium chloride solution (to prevent precipitation of magnesium) and the ammonium carbonate solution and warming(?). The precipitate, if any, is calcium carbonate.

If the results are entirely negative, or if magnesium was suspected in **f**, look for this element [704, par. 4] by dissolving a little of the original substance and trying the test as in Ex. 70 c(?).

h. Report. State your conclusion, with the reasons therefor(?). Also, show that the conclusion is in harmony with the observations recorded under **b** and **c**(?).

j. The Negative Radical in the unknown should now be identified by the plan outlined in §§ 436 and 437, and detailed in Exercises 47 and 63. Report as in **h**.

APPENDIX

I. Tension of Aqueous Vapor.

Temp. C.	Press. mm.	Temp. C.	Press. mm.	Temp. C.	Press. mm.
10	9.2	18	15.4	26	25.1
11	9.8	19	16.3	27	26.5
12	10.5	20	17.4	28	28.1
13	11.2	21	18.5	29	29.8
14	11.9	22	19.7	30	31.5
15	12.7	23	20.9	31	33.4
16	13.5	24	22.2	32	35.4
17	14.4	25	23.6		

II. Apparatus—Individual.

Beakers(2), 100 c.c., 300 c.c.	Funnel, 7 cm.(O.D.*)
Bottle, w.-m. 90 c.c. Mouth to fit No. 5 stopper.	Glass plates(3), 8 cm.
Bottle, n.-m., 2 l.	Glass rod, 2.5 mm.(O.D.)
Bottles(3), w.-m., 250 c.c.	Glass tubing, 6.5 mm.(O.D.)
Bunsen burner.	Graduated cylinder, 50 c.c.
Burette(Mohr's) 25 c.c.	Mortar(porc.), 10 cm.(O.D.), pestle.
Burette clamp(for stand).	Pinchclamps(2).
Cardboard, 10 cm. sq.	Platinum wire(mounted), No. 28, 7 cm.
Crucible(porc.), 25 c.c.	Pneumatic Trough, with shelf or support for bottles. Stoneware milk pan or agate pan about 12 x 4 inches serves admirably.
Deflagrating spoon, small.	Retort(glass stopper), 150 c.c.
Dropper(medicine).	Rubber stopper, No. 5, 2-hole, for wide t.t. and 90 c.c. bottle.
Evaporating dish(porc.), 125 c.c.	
File, triangular.	
Filter paper, cut, 10 cm.	
Flask, 200 c.c. Neck to fit No. 3 stopper.	

* O.D.= outside diameter. I.D.= inside diameter.

Rubber stopper No. 3, 1-hole, for h.g. t.t. and 200 c.c. flask.	Test-tube(1), hard glass, 15 x 2.2 cm.(I.D.)
Rubber stopper No. 2, 1-hole, for t.t.	Test-tubes(6) 15 x 1.8 cm.(I.D.)
Rubber gas tubing, 7 mm. (I.D.), 60 cm.	Test-tubes(12) 10 x 1.2 cm. (I.D.)
Rubber tubing(pure gum), 5 mm.(I.D.) 30 cm.	Test-tube brush.
Stand, rod 50 cm., 1 ring 9-10 cm.	Test-tube rack.
Taper.	Thistle tube, small.
Test-tube(1) 15 x 2.5 cm.(I.D.)	Triangle(pipe stem).
	Watch glasses(2), 5 cm., 8 cm.
	Wing top(for burner).
	Wire, iron, No. 26, 20 cm.
	Wire gauze, iron, 10 cm.

III. Apparatus—General.

Balance. Load 100 g., agate planes and agate knife edges, in glass case, without rider- beam(cost \$15, duty free).	Meter stick.
Barometer.	Paper. Roll, smooth, colored (to prevent use for writing).
Bottles, square, 25 c.c.(1:5*).	Pipettes, 20 c.c.(1:10).
Cobalt glasses, 10 x 5 cm.(1:5).	Pliers, cutting.
Corks to fit test-tubes(1.8 cm. I.D.), 2 l. bottle, and w.-m. bottles.	Thermometers, — 10° to 150° (1:5).
Cork-borers(1:5).	Thread.
File, round.	Trip scales with rider(0-5 g.) and weights(5 g.—1000 g.)
Graduated cylinders, 500 c.c. (1:10*).	Waste jars(1:4).
Kipp's generator.	Weights. Good grade labora- tory weights, 1 mgrm. to 50 g., fractional weights in divided compartments under glass cover, all in wooden block with hinged cover.
Lens(coddington).	
Magnet.	

IV. Materials. (*All included.*)

Acid, acetic.	Acid, hydrochloric, conc.
formic.	nitric, conc.

* Signifies 1 to 5 pupils, 1 to 10 pupils, etc.

Acid, orthophosphoric.

oxalic.

pyrogallie.

sulphuric, conc.

tannic.

Acid Green (or Eosin).

Albumen, dry com'l. egg.

Alcohol, amyl.

ethyl, 95%.

Alizarin paste (20%).

Almonds.

Aluminium, wire.

Aluminium sulphate.

Ammonium carbonate.

chloride.

hydroxide, com'l.

molybdate.

nitrate.

oxalate.

sulphate.

sulphide.

Antimony trichloride.

Apples.

Arsenic trioxide.

Asbestos, long fiber.

paper.

wool.

Barium chloride.

hydroxide.

Benzene.

Bismuth nitrate.

Bleaching powder.

Borax.

Bromine.

Calcium.

Calcium carbide.

carbonate (marble chips).

Calcium, carbonate (pulv.)

chloride (gran.)

oxide (quicklime).

phosphide.

sulphate (gypsum).

sulphate (pulv.)

Carbon disulphide, com'l.

tetrachloride.

Charcoal, bone (pulv.)

Charcoal, wood (splinters)

wood (pulv.)

Chromic oxide.

Chrysophenin.

Clay.

Cloth, cheese.

colored calico.

white cotton.

white flannel.

white mixed goods.

Coal, bituminous.

Cobalt chloride.

Copper, foil.

shavings.

wire, No. 30.

Cotton seed oil.

Cupric oxide (pulv.)

sulphate

Eosin (or Acid Green).

Ether, ethyl.

Fat.

Ferric chloride.

oxide (pulv.)

Ferrous chloride.

sulphate.

sulphide.

Flour.

Formaldehyde (sol.)

- Gasoline.
 Gelatine(flakes).
 Glucose, brewers, *cryst.*
 Graphite.
 Iodine.
 Iron, nails.
 powder(by alcohol).
 wire, No. 20.
 Kerosene oil.
 Labels.
 Lead, sheet.
 Lead dioxide.
 monoxide.
 nitrate.
 Lithium chloride.
 Litmus paper, blue and red.
 sol., neutral.
 Logwood.
 Magnesium, ribbon.
 wire.
 Magnesium carbonate(pulv.)
 chloride.
 Malachite Green.
 Manganese dioxide(pulv.)
 Matches.
 Meat.
 Mercurous chloride.
 nitrate.
 Mercury.
 Methyl acetate, com'l.
 Methyl Violet.
 Milk.
 Molasses.
 Nickel, powder(reduced).
 Nickel sulphate.
 Paraffin
 Phenolphthalein.
 Phosphorus, red.
 Phosphorus, white.
 Pinks, or grass or leaves.
 Potassium bitartrate.
 bromide.
 chlorate.
 chloride.
 chromate.
 dichromate.
 ferricyanide.
 ferrocyanide.
 hydroxide.
 iodide.
 permanganate.
 sulphate.
 sulphocyanate.
 Potassium antimonyl tartrate
 (tartar emetic).
 Potassium-sodium tartrate(Ro-
 chelle Salt).
 Rosin(pulv.)
 Rubber bands(small).
 Sand.
 Sausage.
 Silver nitrate.
 Soap, castile.
 ivory.
 Soda lime.
 Sodium acetate.
 bicarbonate.
 bisulphate, com'l.
 bisulphite.
 carbonate(anhyd.)
 carbonate(cryst.)
 chloride.
 hydroxide.
 nitrate.
 peroxide.
 phosphate.

Sodium, sulphate (cryst.)	Vinegar (white).
Stannic oxide.	Wood, sawdust and chips.
Starch.	splints (tobacconists').
Strontium chloride.	Woolen yarn.
Sugar, milk.	Yeast.
Sugar, ordinary (sucrose).	Zinc, dust.
Sulphur, flowers.	gran.
roll.	sheet.
Tin, foil (lead free).	Zinc oxide.
gran.	

V. Apparatus and Materials—Optional Exercises.

Bottles, n.-m., 1 liter (Ex. 25).	Iron, piano-wire (Ex. 25).
Bottles, tall form, 500 c.c. (Ex. 68).	Zinc, C. P. (Ex. 25).

VI. Unknowns Suggested for Exercises 47, 63 and 87. (*All solids.**)

Exercise 47. Ammonium carbonate, chloride, and sulphate.

Sodium acetate, carbonate, chloride, formate, hydroxide, sulphate, sulphide, and sulphite.

Potassium carbonate, chloride, and sulphate.

Calcium carbonate, chloride, hydroxide, and sulphide.

Starch, sucrose, glucose (crystal), and sulphur (pulv.).

Exercise 63. Barium peroxide.

Ammonium carbonate, nitrate, and phosphate.

Sodium bicarbonate, bisulphite, carbonate, chloride, nitrate, peroxide, phosphate, and sulphite.

Potassium bromide, iodide, and nitrate.

Calcium bisulphite and carbonate.

Exercise 87. Any compounds of the metals listed, except insoluble compounds of calcium or magnesium.

* To be added, where necessary, to the general list of materials (Appendix IV).

VII. Chapters and Corresponding Exercises.

The Roman numerals refer to chapters in the Author's *Text-book of Elementary Chemistry*, and the Arabic numerals to the laboratory exercises.

CHAP- TER	EXER- CISES	CHAP- TER	EXER- CISES	CHAP- TER	EXER- CISES
I	1, 2, 3	XV	25	Review	60
II	4, 5	XVI	28	XXX (Review)	63
IV	6, 7, 8	XVII	29, 30, 31	XXXI	64, 65, 66
V	14	XVIII	32, 33	XXXIII	67, 68, 69
VI	9, 10	XIX	34, 35, 36	XXXIV	70
VII	11, 12	XX	37, 38	Review	71
VII or	13	XXI	39, 40	XXXV	72, 73, 74, 75
VIII		XXII	41, 42, 43	XXXVI	76, 77, 78, 79, 80
VIII	14, 15	XXIII	44, 45, 46	XXXVII	81
VIII or	16, 17	Review	47	XXXVIII	82
IX		XXIV	48, 49	XXXIX	83, 84
X	18, 19	XXV	50, 51, 52	Review	85
XI	20	XXVI	53, 54, 55	XLI	86
XII	21	XXVII	56, 57	XLII (Review)	87
XIII	22	XXVIII	58		
XIV	23, 24, 26, 27	XXIX	59, 61, 62		

**This book is under no circumstances to be
taken from the Building**

[illegible]

